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Very strong nanometals and nanostructured surfaces

Juul Jensen, Dorte

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Very strong nanometals and nanostructured surfaces

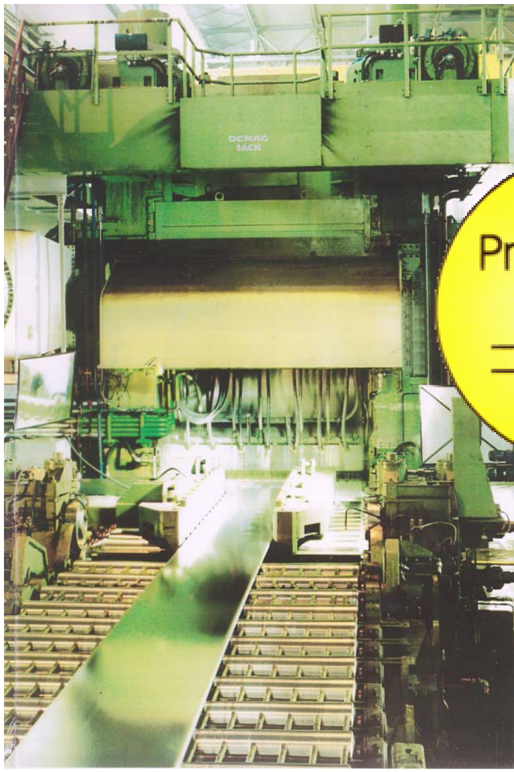
D. Juul Jensen

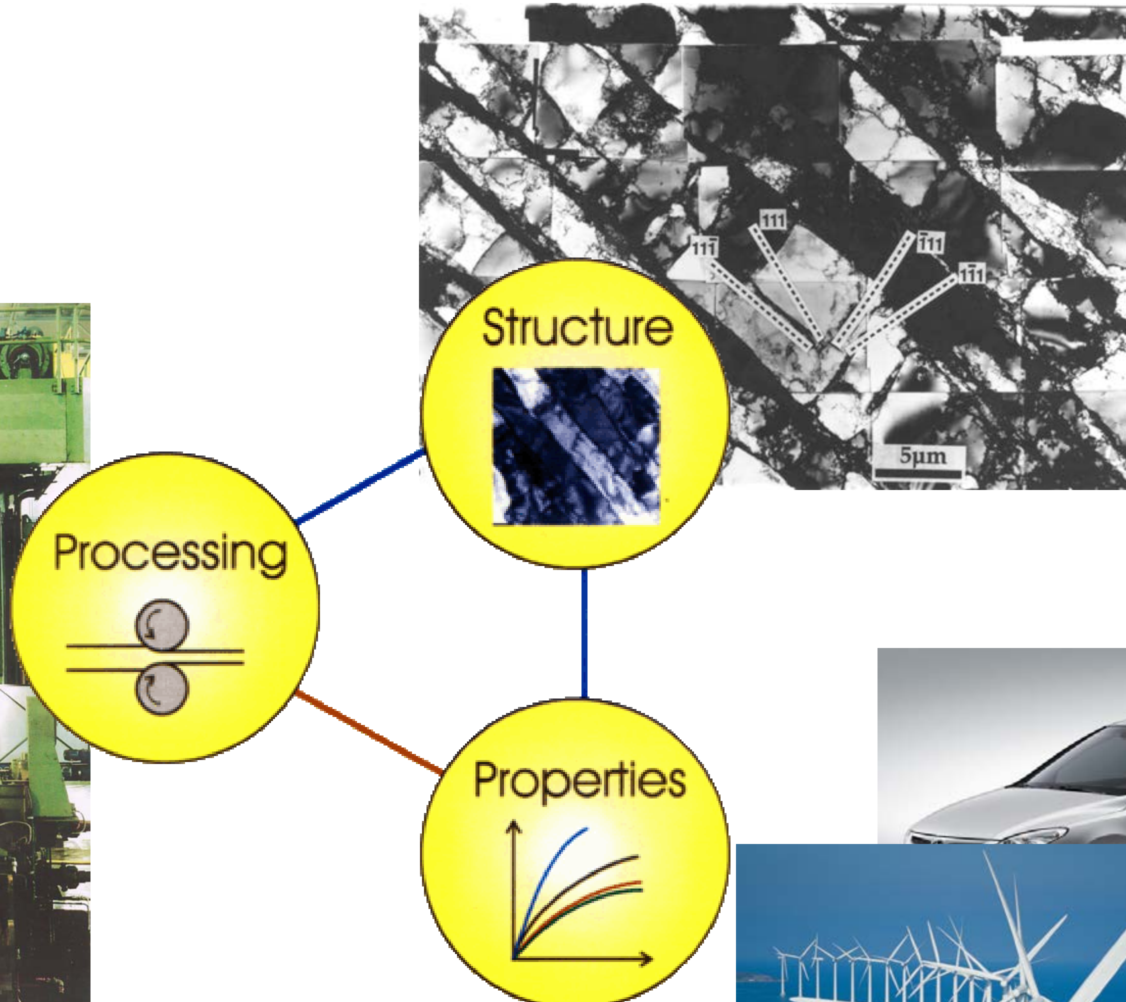
DTU Wind Energy
Department of Wind Energy

Section:
Materials Science and Advanced Characterization

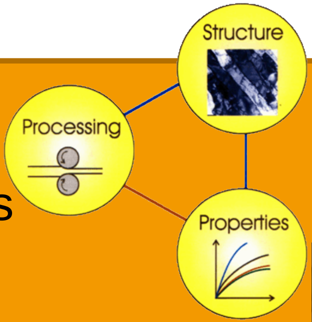
1 Professor
3 Senior researchers
4 Researchers
2 Post Docs
3 PhD students
4 Technicians
1 Secretary
2 Emeritus

Very close collaboration with the section: Composite Materials and Mechanics




Structure
5μm
Processing
Properties

Materials:
Light and strong metals and alloys
Steels
Nanostructured materials



- <u>Processing</u>
  Rolling, extrusion, etc.
  Very high strain: ARB, DPD HPT
  Annealing

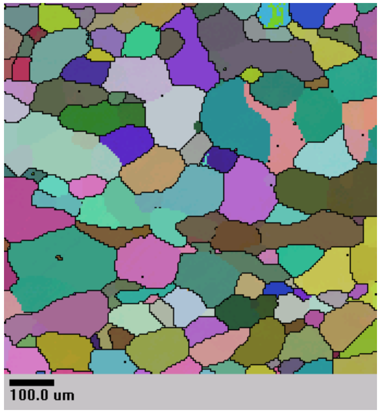
- <u>Structure</u>
  Advanced electron microscopy
  Advanced x-ray characterization
  Advanced sample preparation
  Serial sectioning

- <u>Properties</u>
  Mechanical testing (KOM)
  Calometry
  Hardness
  Physical proporties

Research

- Hard and wear resistant steel components
- Light and strong metals and alloys
- Technique development

Electron microscopes @ DTU Wind Energy

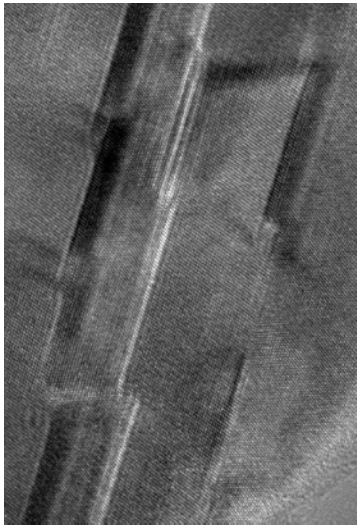


3 SEM & 3TEM

ZEISS SUPRA 35

JEOL JMS-840

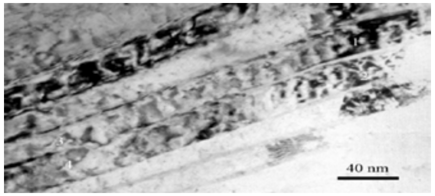
ZEISS EVO 60

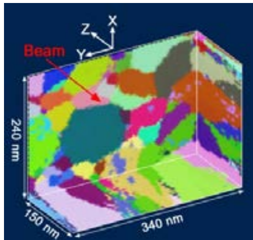


JEOL JEM-3000F

JEOL JEM-2000FX

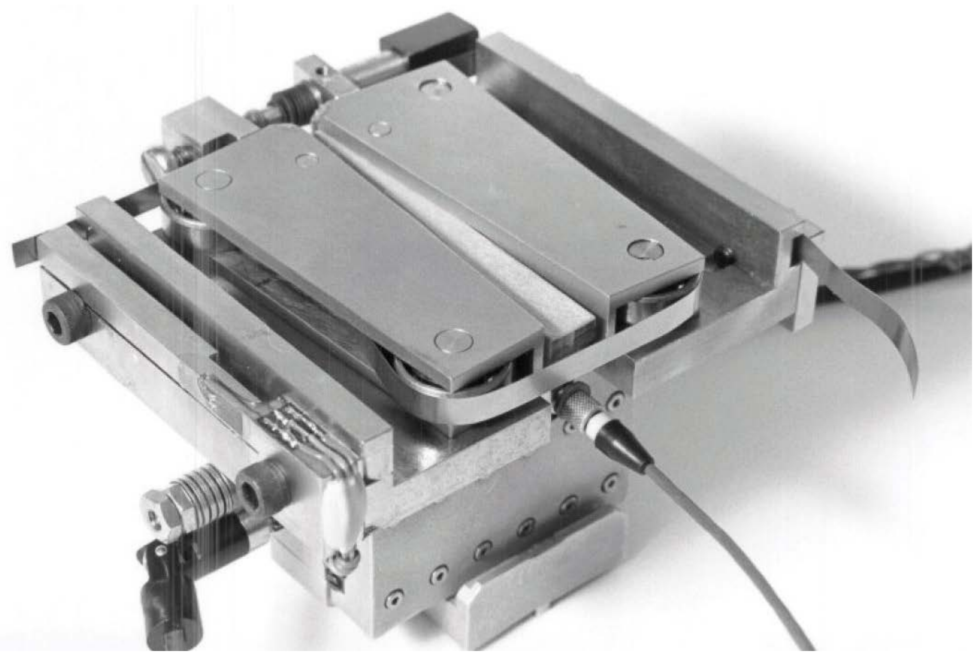
JEOL JEM 2100

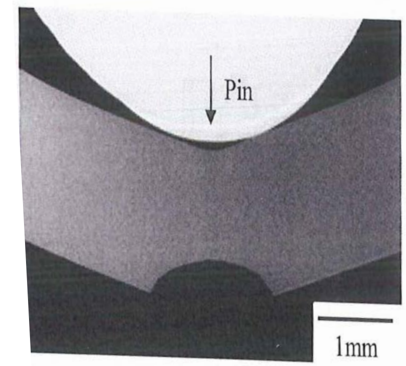




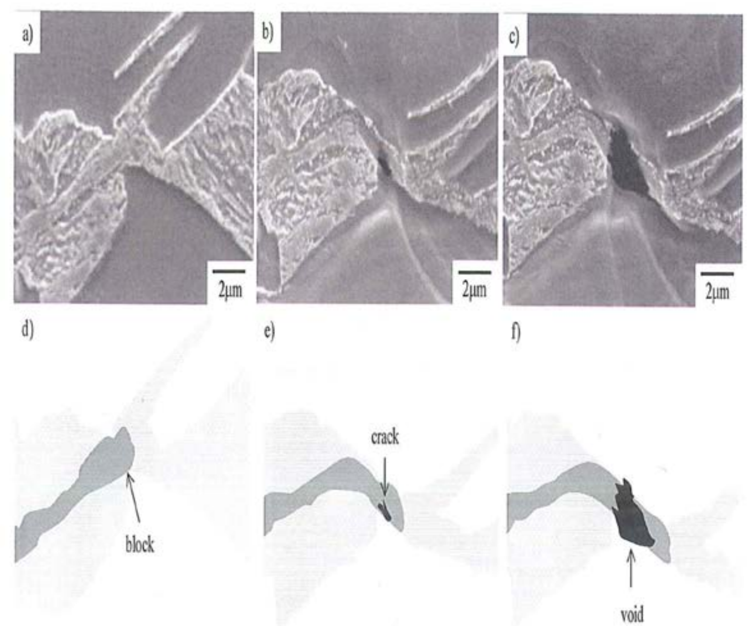
Mechanical test fixtures i ESEM and HR TEM

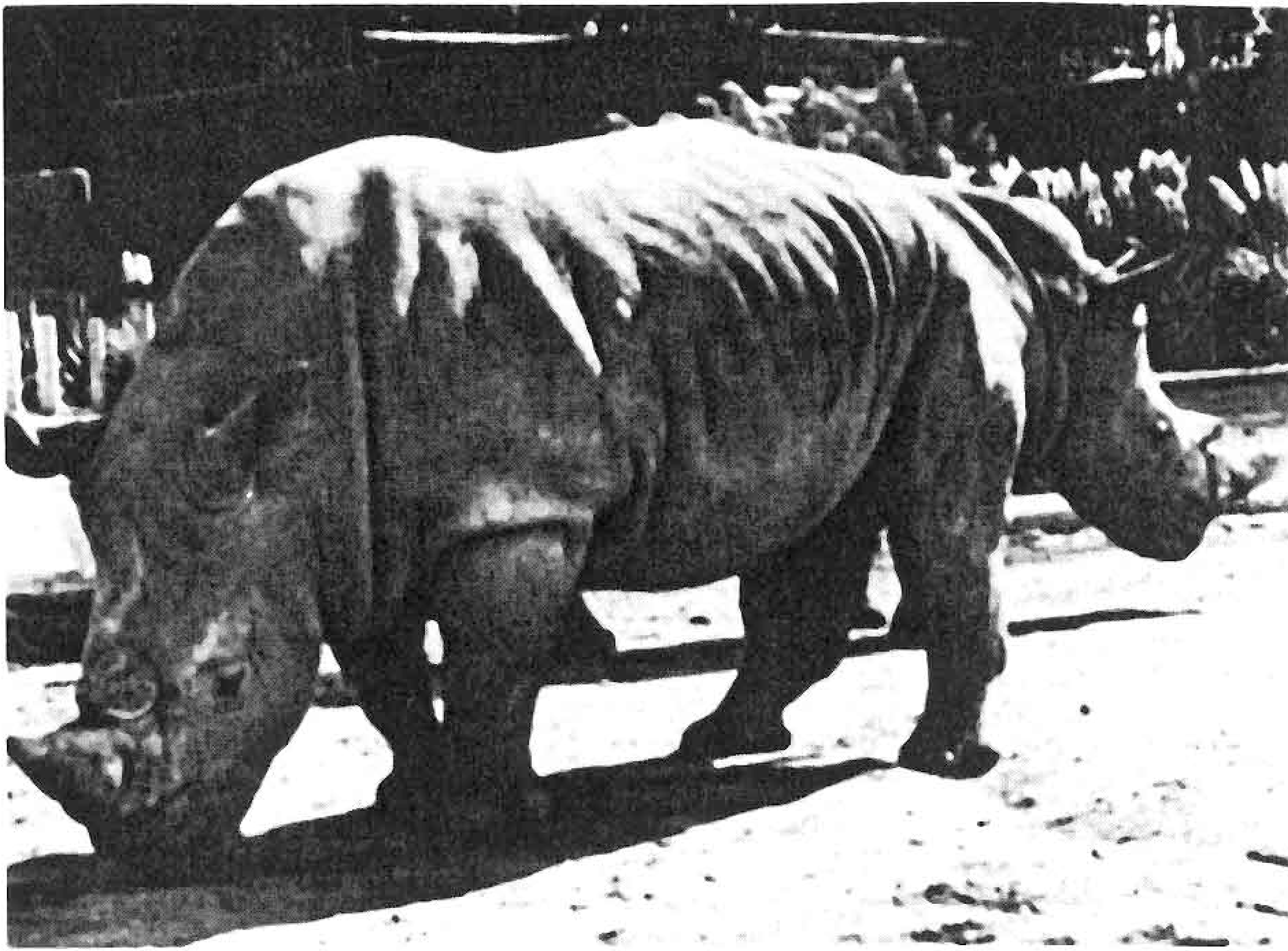
– in-situ observations of failure mechanisms





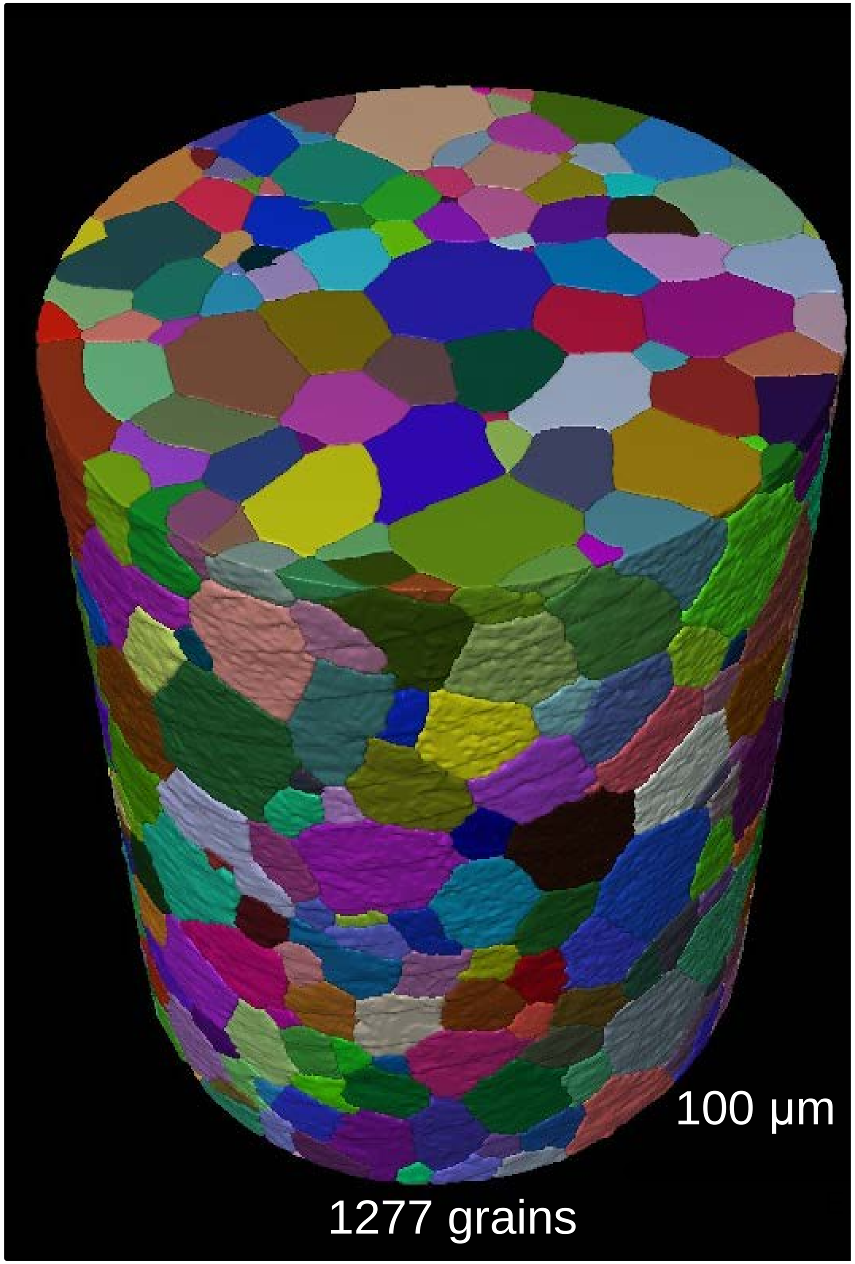
During deformation

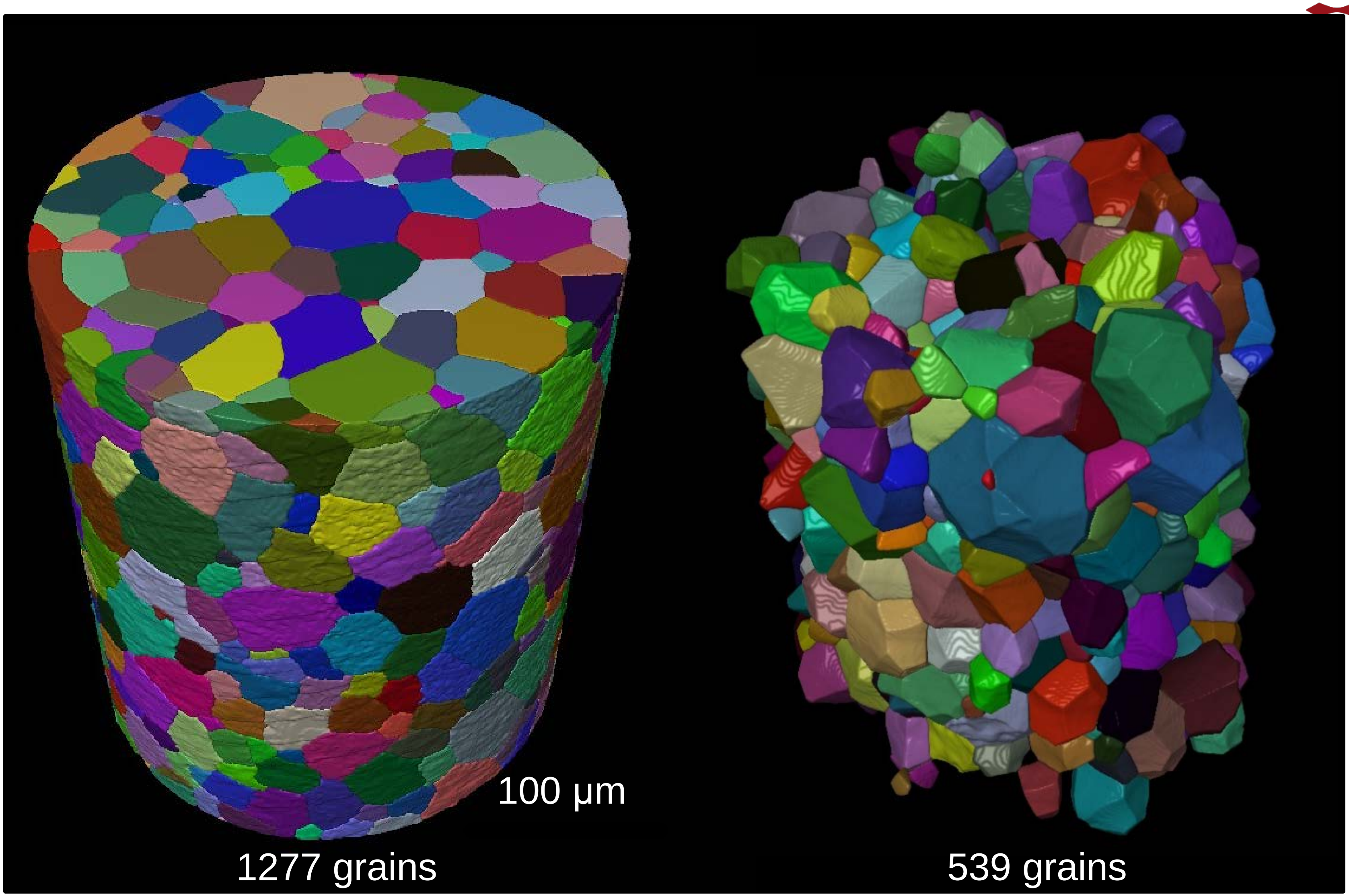




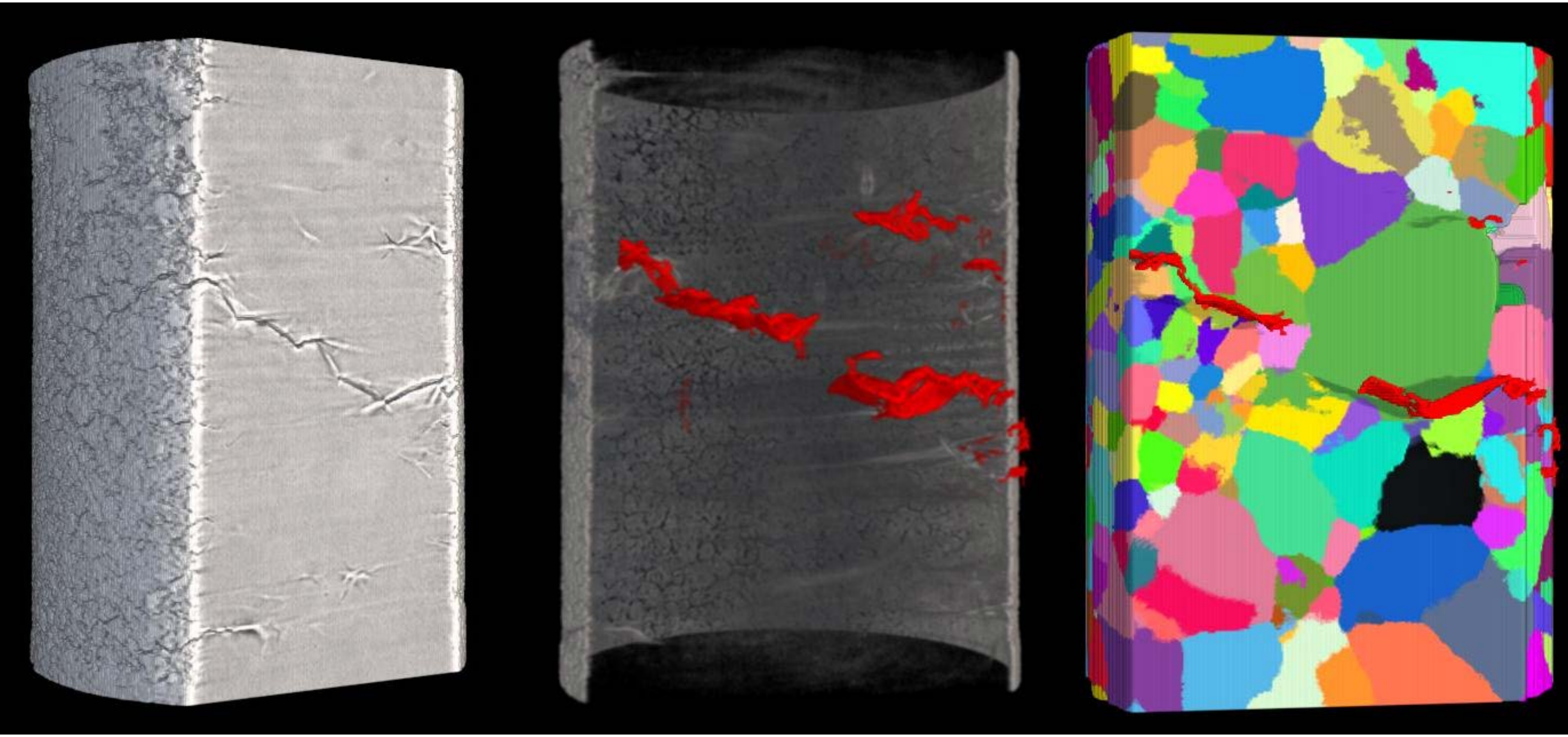
3D x-ray microscope for in-situ characterization

- μm spatial resolution
- Bulk penetration (0.1 mm – 1cm)
- Non-destructive
- Fast measurements (seconds – minutes)

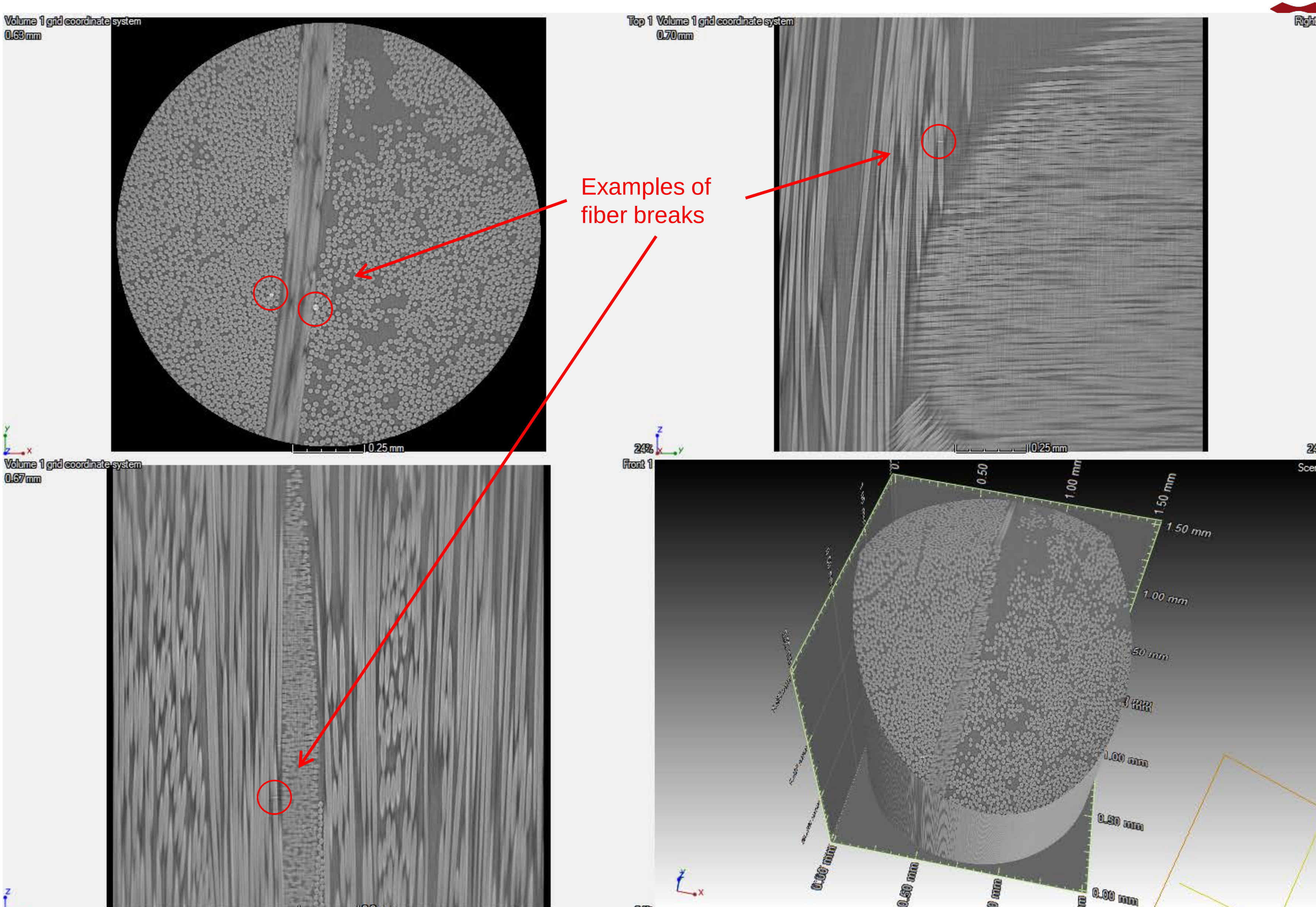

100 µm
1277 grains



E.M. Lauridsen and S.O. Poulsen



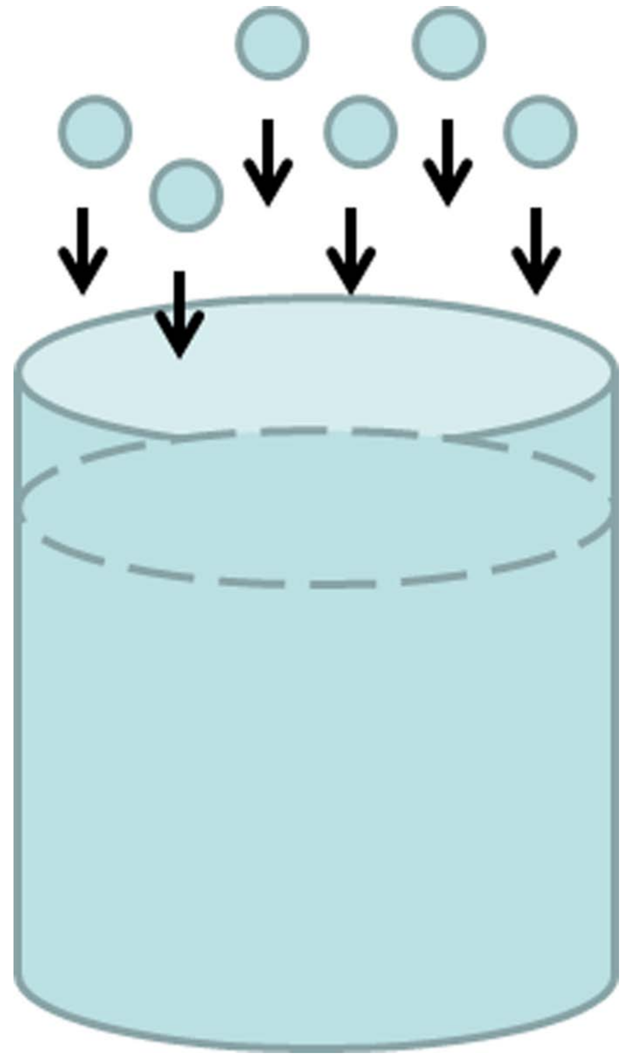
A. King, G. Johnson, D. Engelberg, W. Ludwig, and J. Marrow,
Science (2008) **321**, 382 - 385


Volume 1 grid coordinate system
0.63 mm
0.25 mm
Top 1 Volume 1 grid coordinate system
0.70 mm
Right 1
0.25 mm
24%
Examples of
fiber breaks
Volume 1 grid coordinate system
0.67 mm
0.2 mm
Front 1
Scene
0.50
1.00 mm
1.50 mm
1.50 mm
1.00 mm
0.50 mm
0.00 mm
24%

Hard and strong surfaces

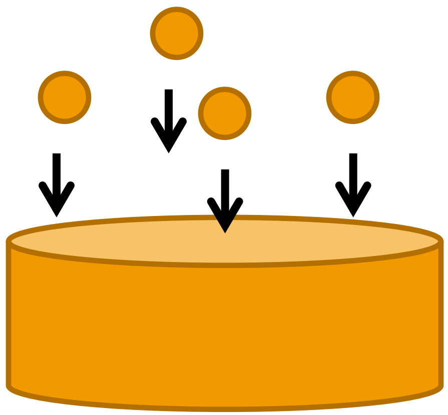
High energy shot peening

High energy shot peening was carried out in air one low carbon steel with 0.8 mm diameter high carbon steel balls (Fe–0.91 C–0.61 Si–0.6 Mn–0.021 P–0.018 S (wt.%), HRC 62). The shot velocity was 260-300 ms^{-1}.

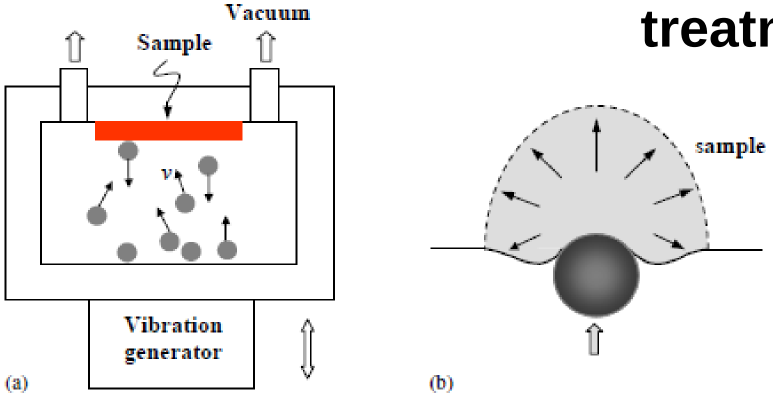


Particle impact

Shot-peening

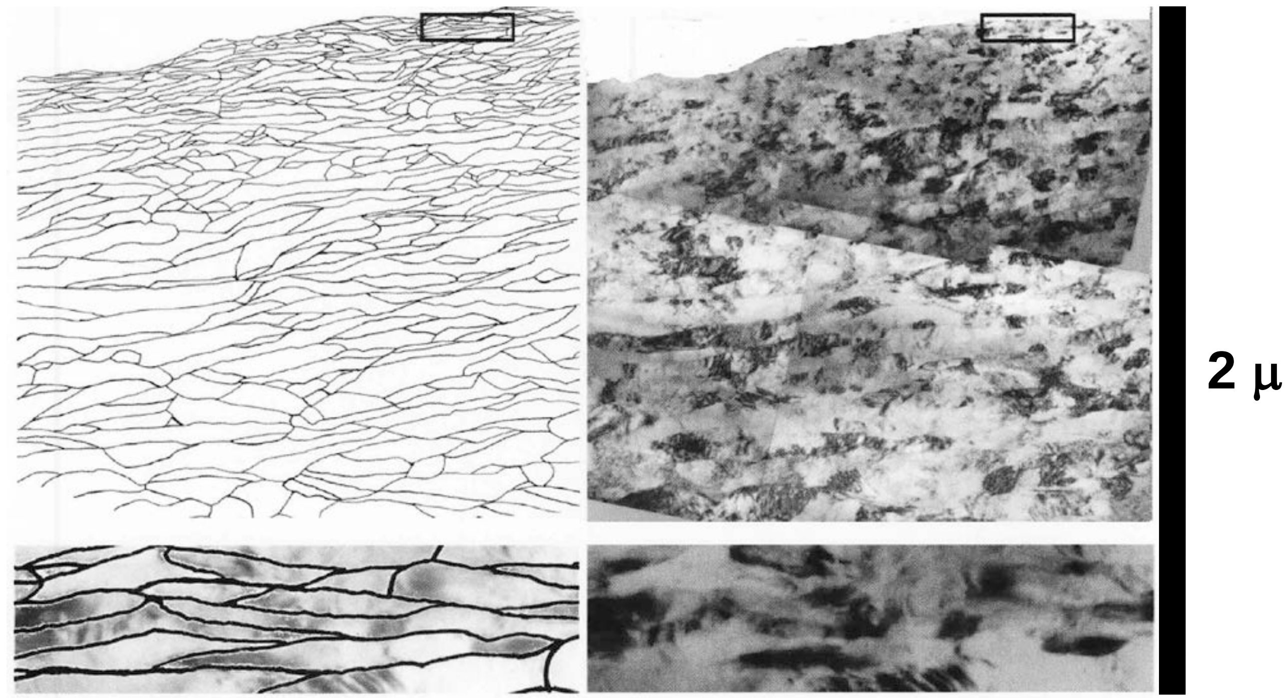


SMAT (surface mechanical attrition treatment)

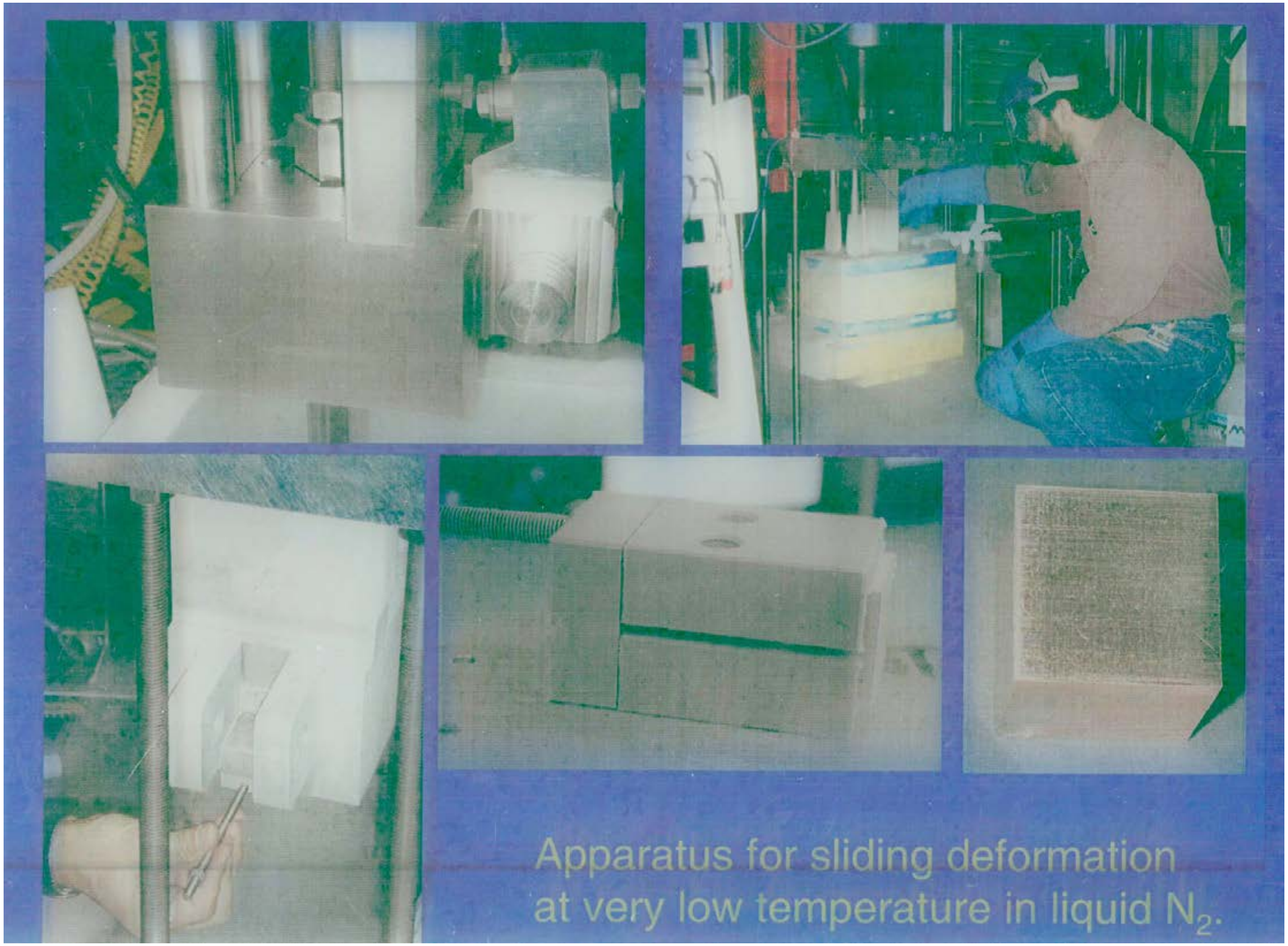


	Shot-peening	SMAT
Shot size	0.05 ~ 1 mm	1 ~ 10 mm
Shot velocity	~ 100 m/s	1 ~20 m/s
Shot direction	Single direction (~ 90°)	Multi-direction (vibration frequency: 20 ~ 50 HZ)
Temperature increase	50-100 ℃	50-100 ℃
Thickness of graded nanostructures	~ 20 μm	~ 40 μm

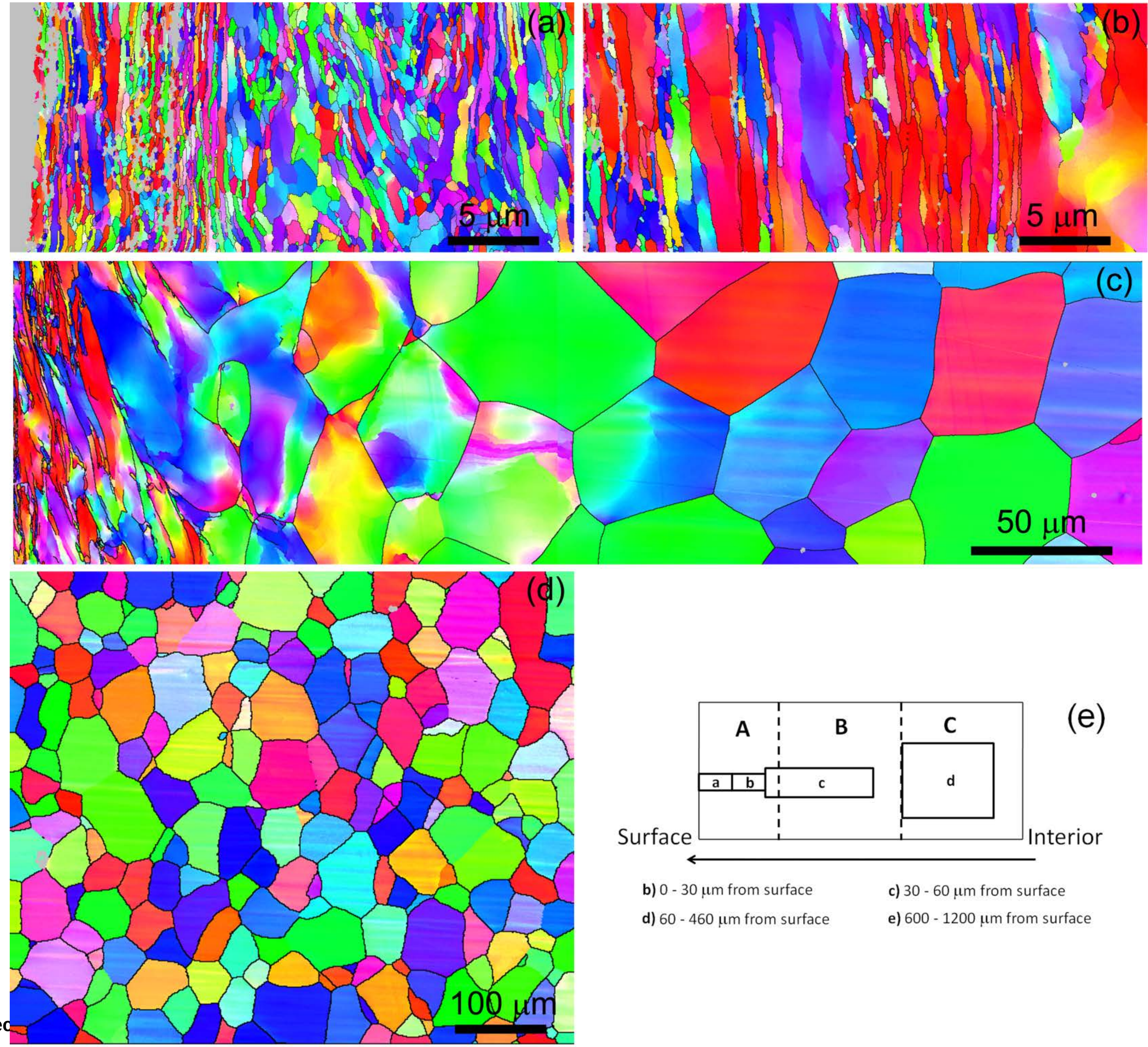
Graded nanostructures produced by sliding enable characterization of the structural scale from 10 to 10,000nm.



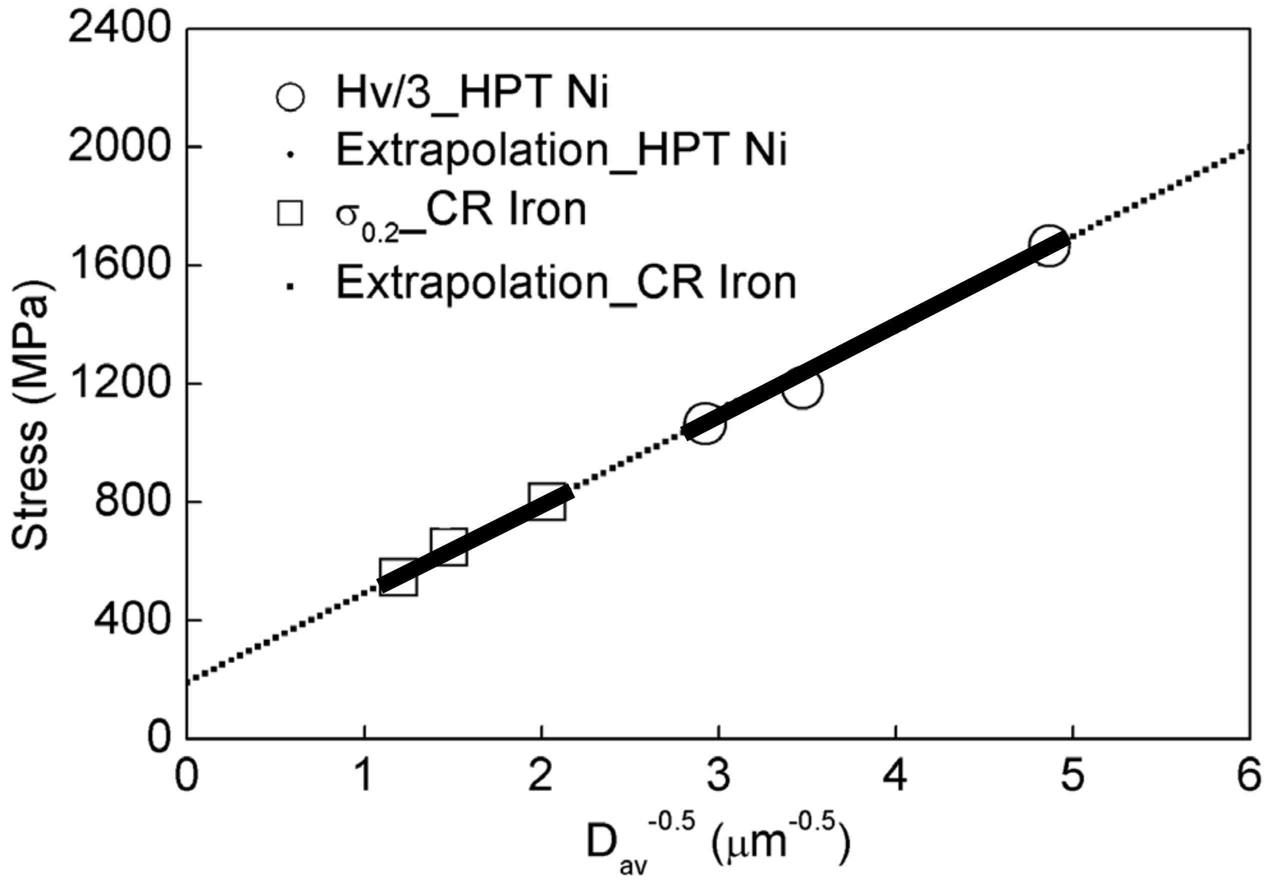
Graded nanostructure in Cu produced by127mm friction deformation under 12MPa viewed in cross-section by TEM, with schematic.


Apparatus for sliding deformation
at very low temperature in liquid N2.

Microstructure Characterizations

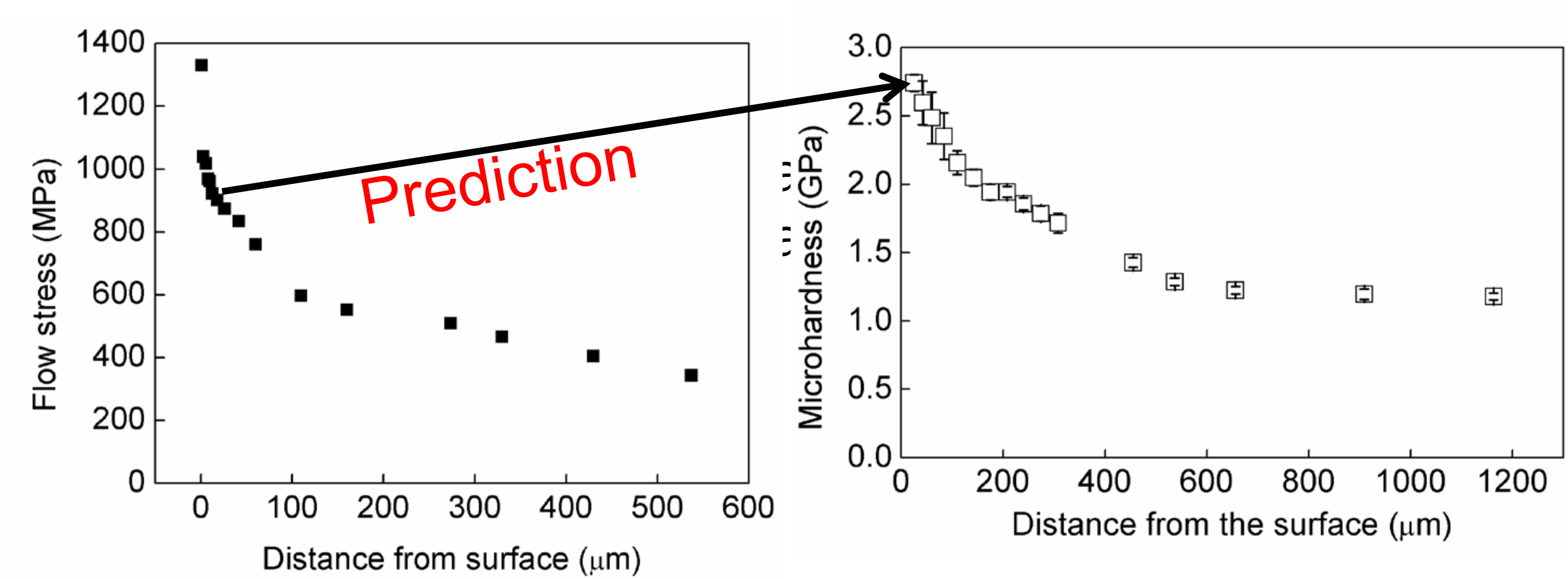


Flow stress (σ) as a function of boundary spacing (D_{av})

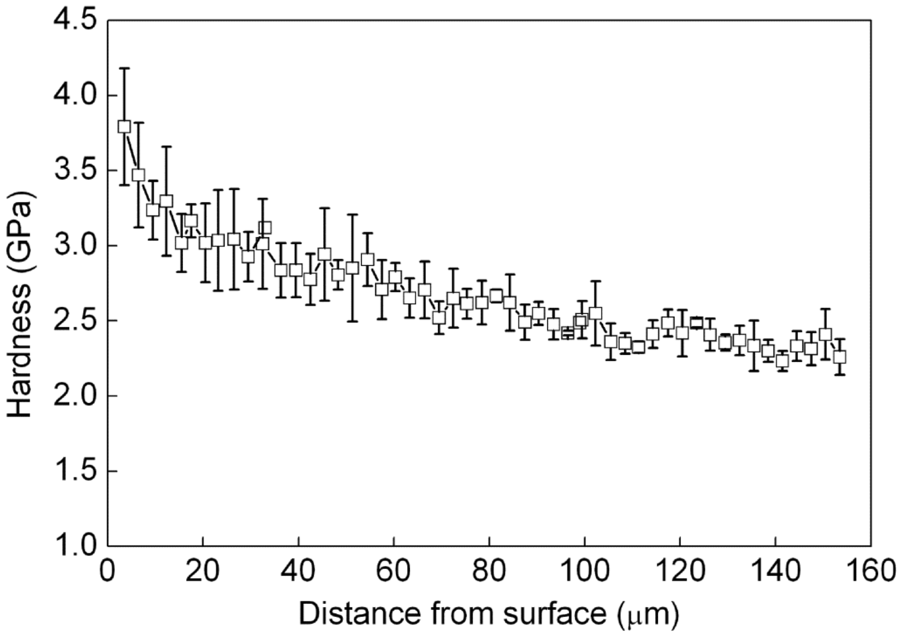


Stress distribution in a graded shot peened surface layer

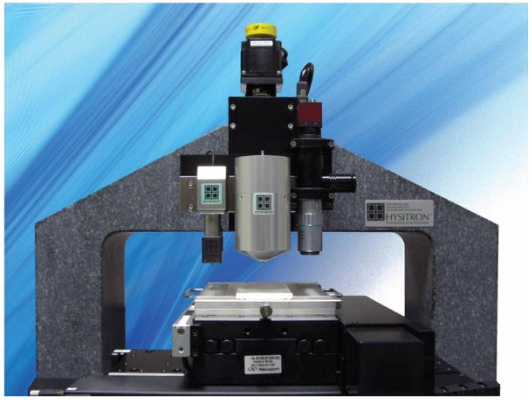
❑ Determined directly by micro hardness (minimum distance from surface about 25 µm)
❑ Determined indirectly based on a master curve for the relationship between σ and D_{av} for bulk samples

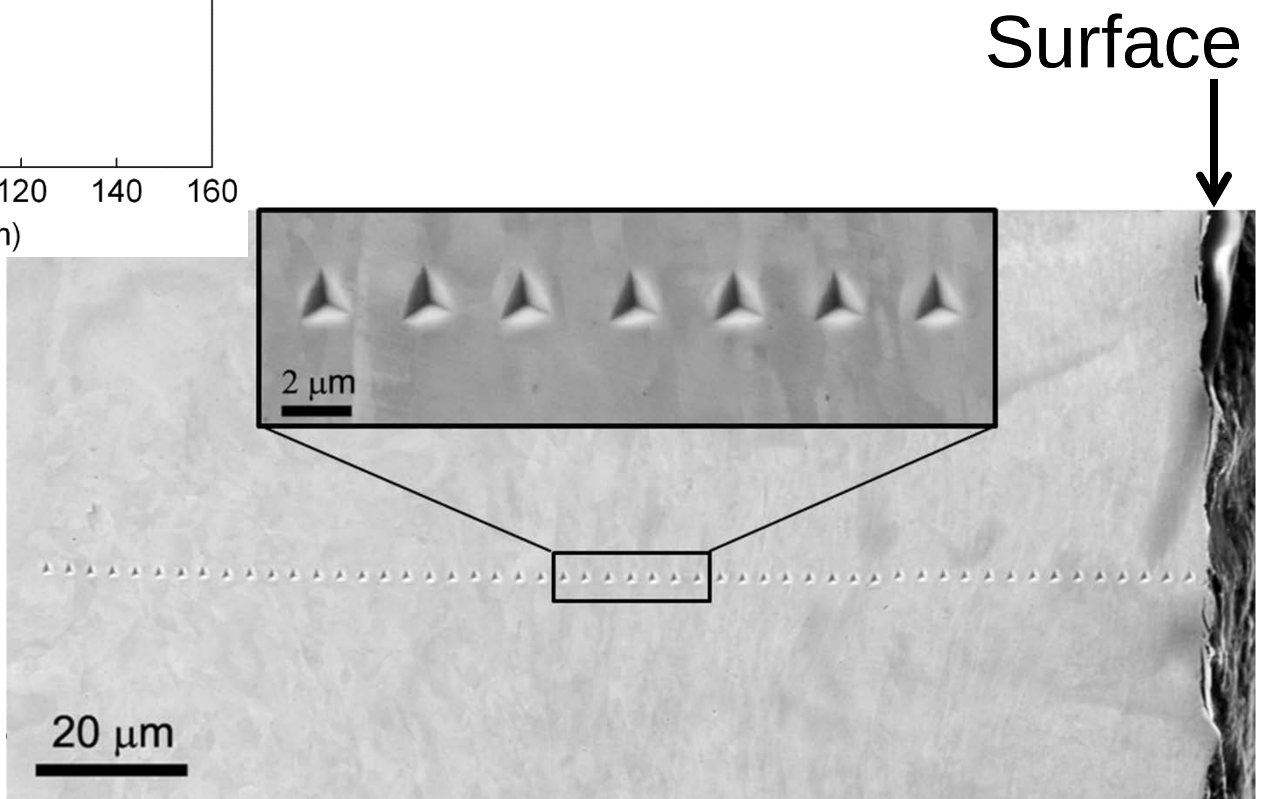


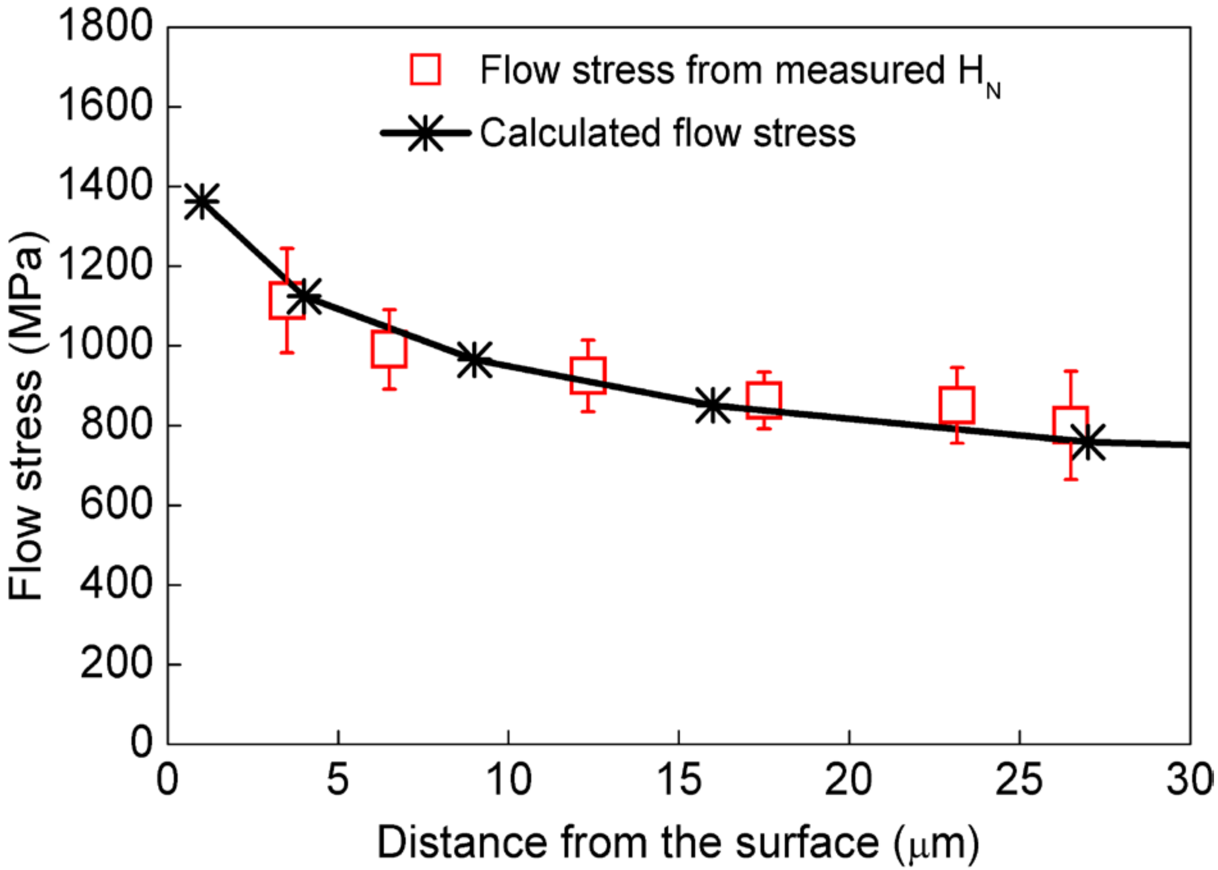
Nanohardness



Nanoindentation has been introduced which allows residual hardness values to be obtained to a depth a few micronmetres below the surface.





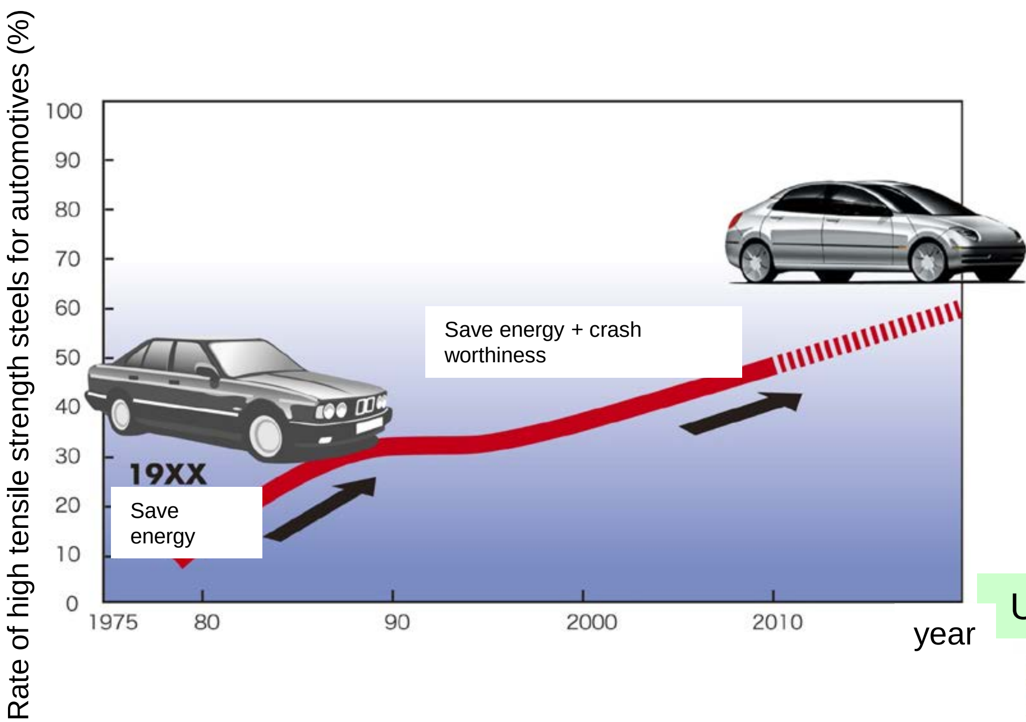


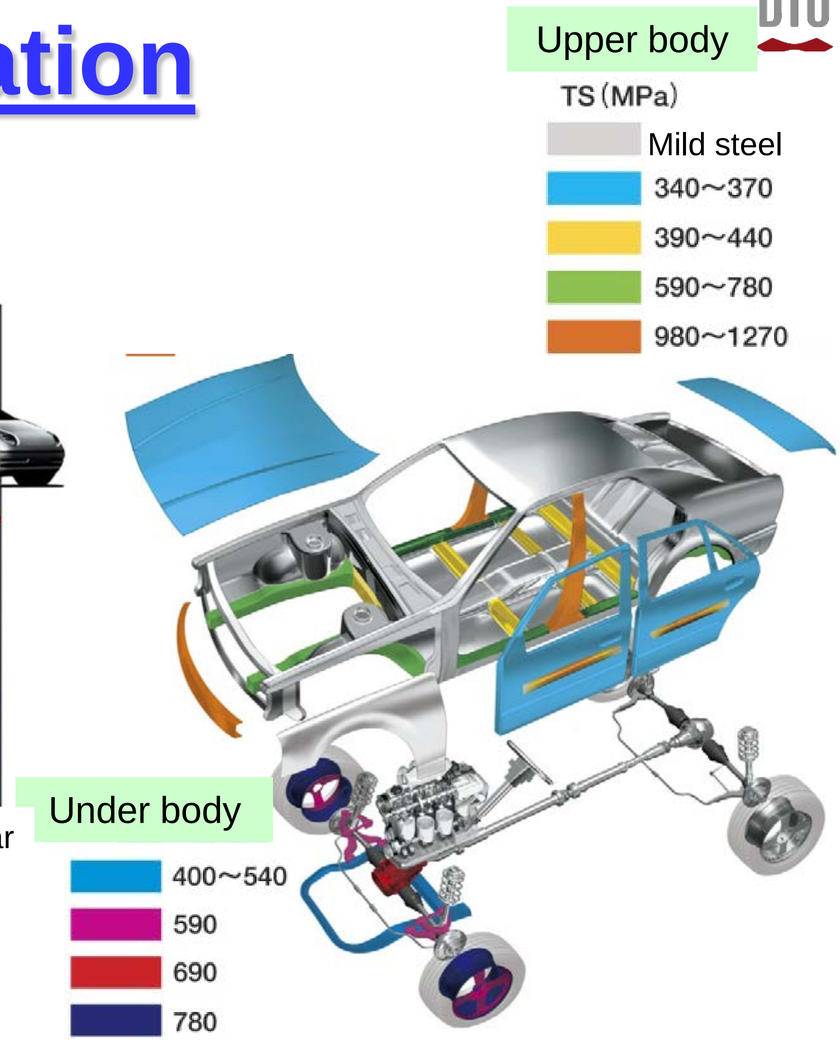
Calculated flow stress and transformed flow stress from nanohardness versus distance from the surface.

Applications

Structure control and void formation in dual phase steels

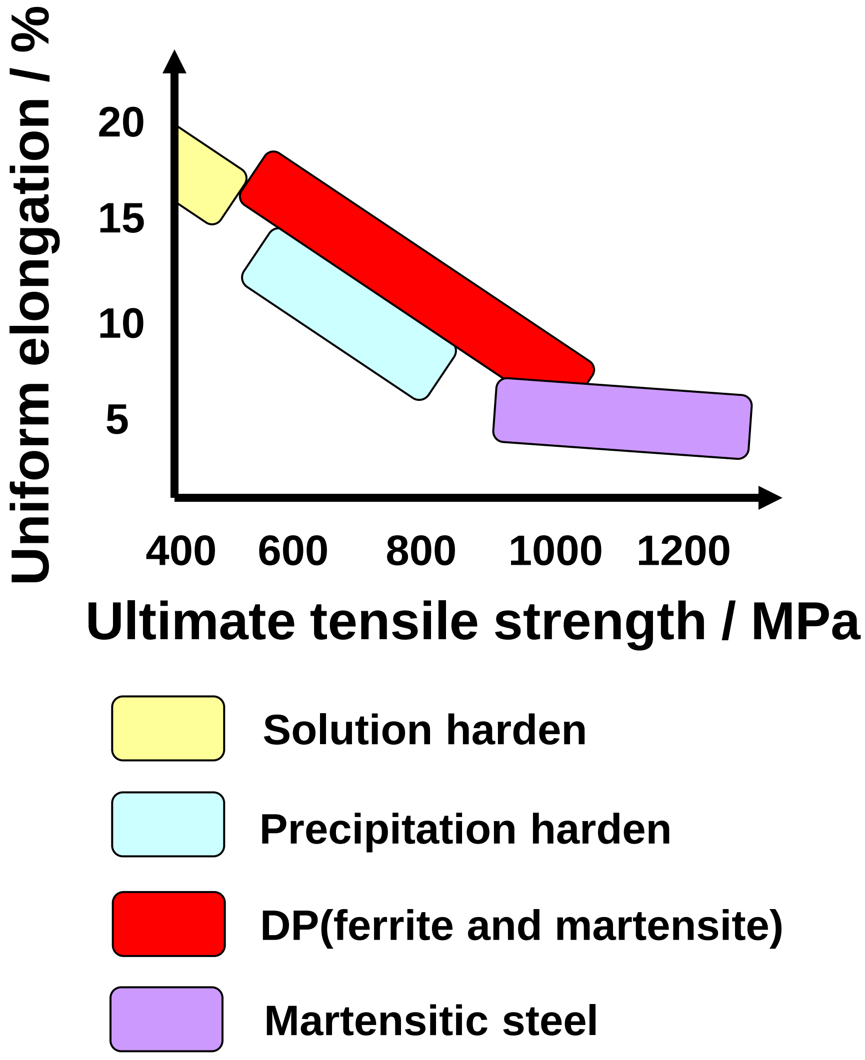
Motivation



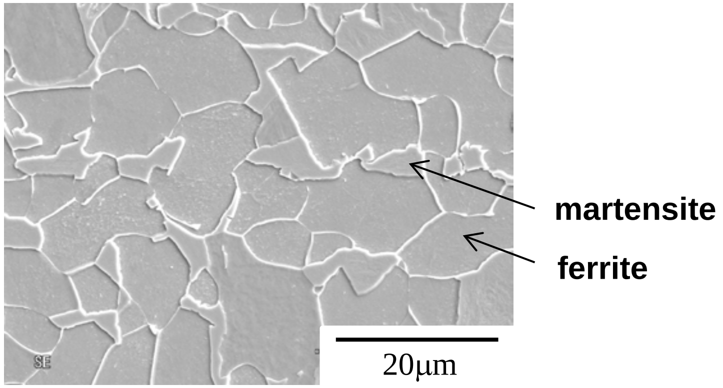


Higher tensile strength steels are often applied for automobiles.

Background 1
-Mechanical properties of high strength steels



DP(ferrite and martensite)



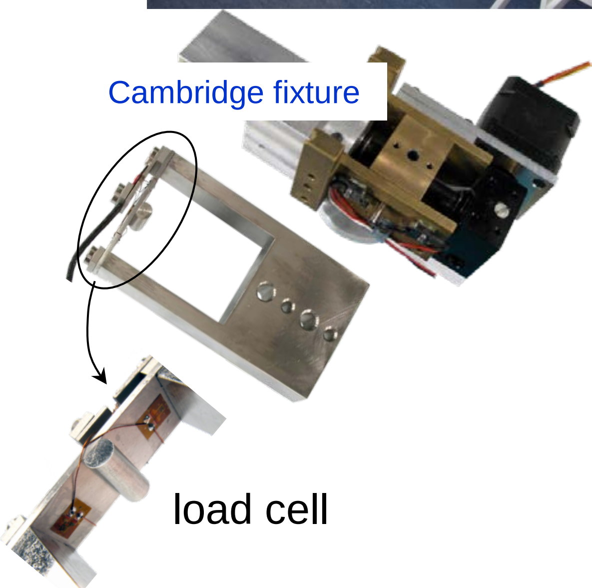
- Ferrite grains ensure uniform elongation.

- Martensite particles give strength.

Excellent combination of ultimate tensile strength and uniform elongation

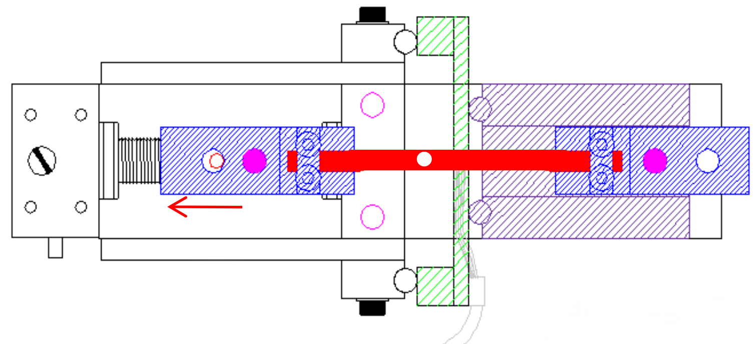
Tensile test in a SEM

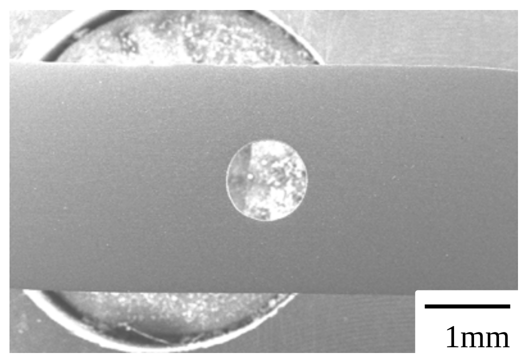
➢ Scanning Electron Microscope (SEM, EVO60)



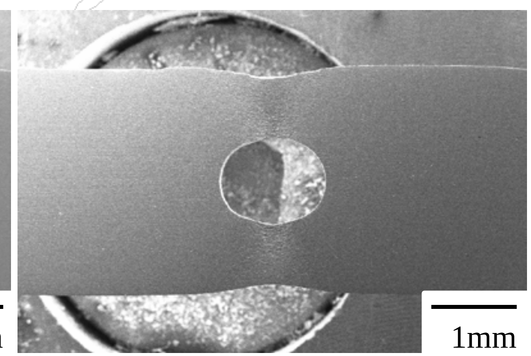
Tensile test

Sample size : 0.15 mm × 2.8 mm × 30 mm
Hole diameter : 1 mm



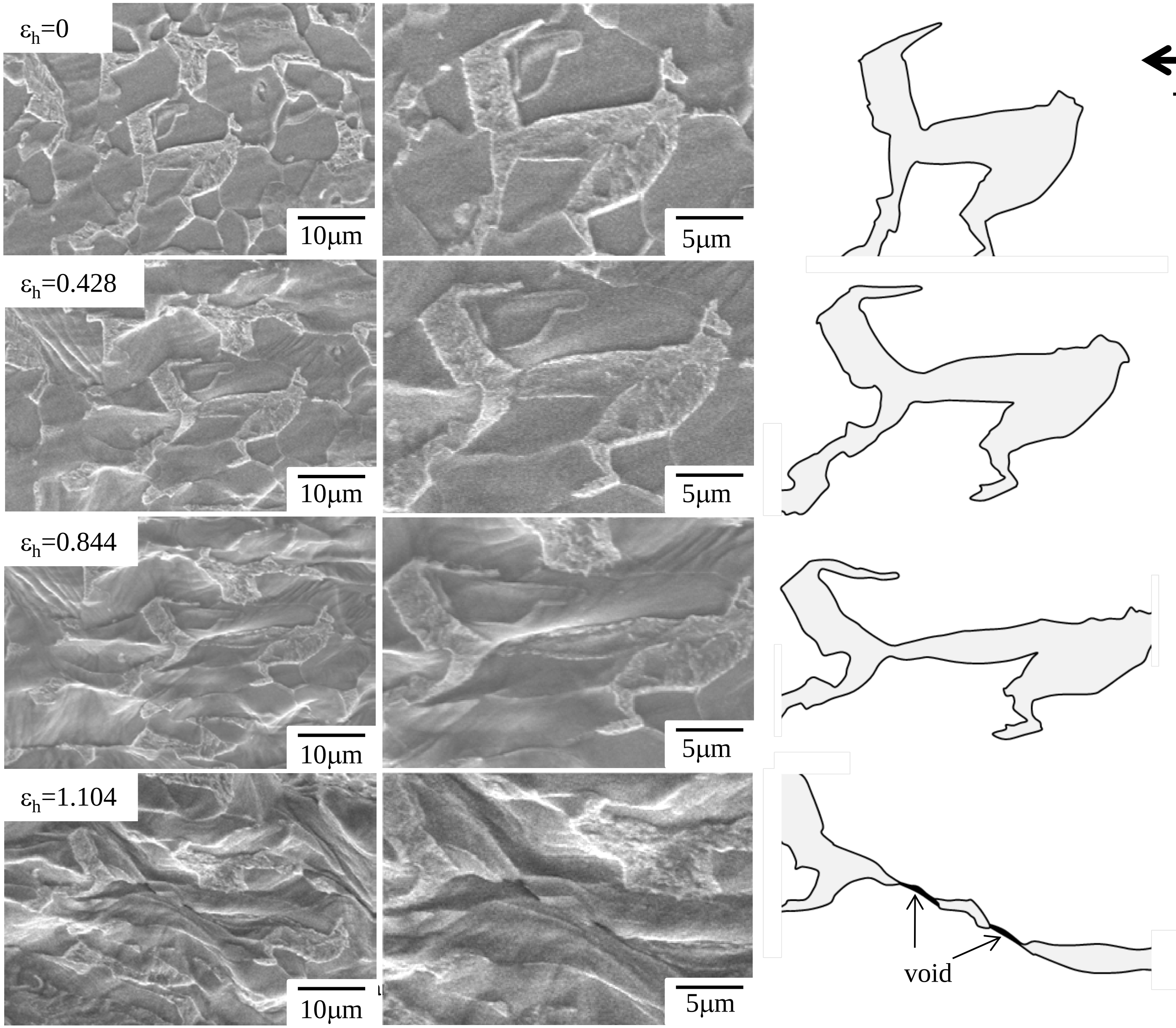


Before a tensile test

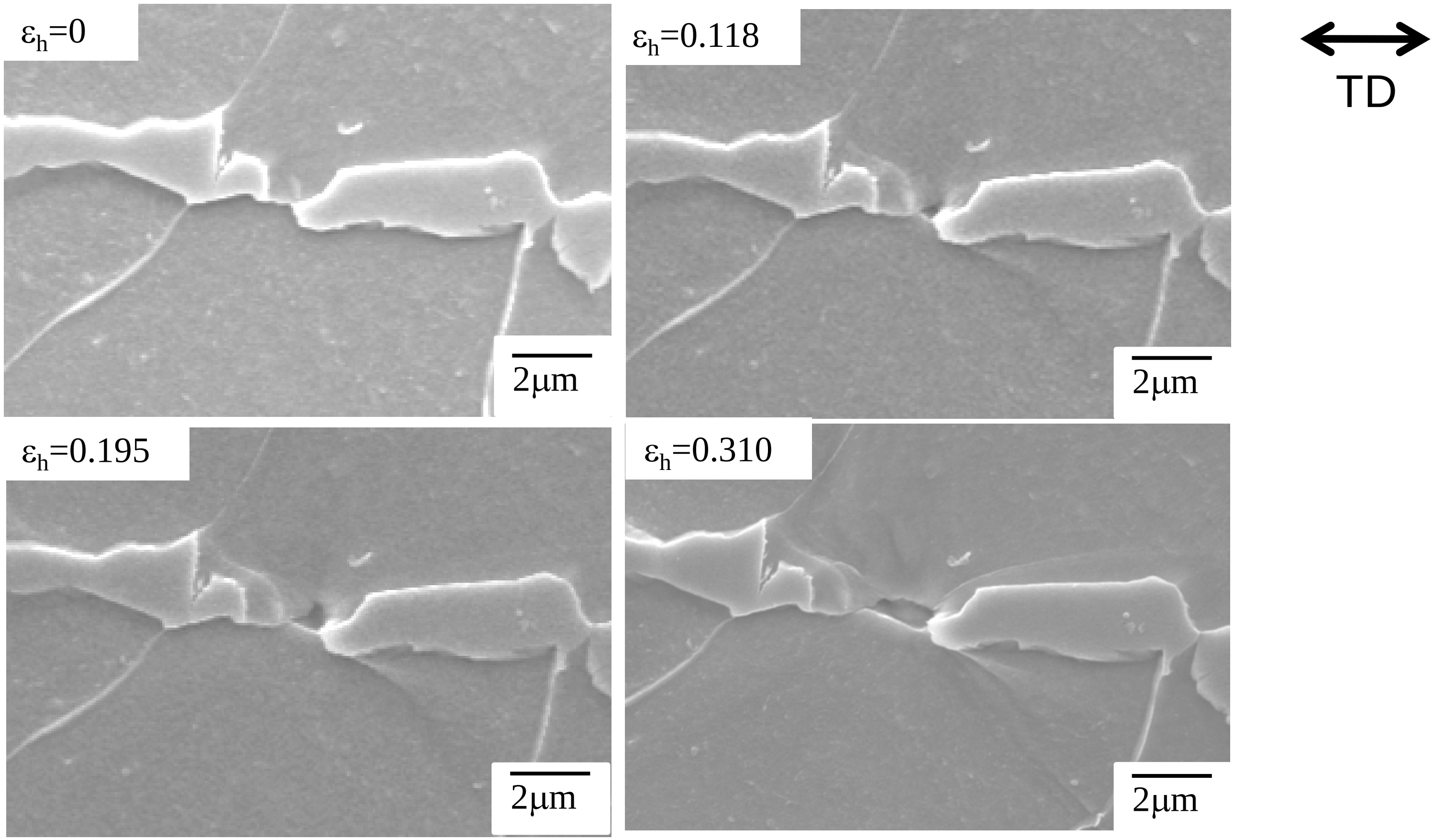


During a tensile test

Void formation after tempering at 500°C.



Void formation behavior at ferrite/martensite interface during in-situ tensile test



Summery of project

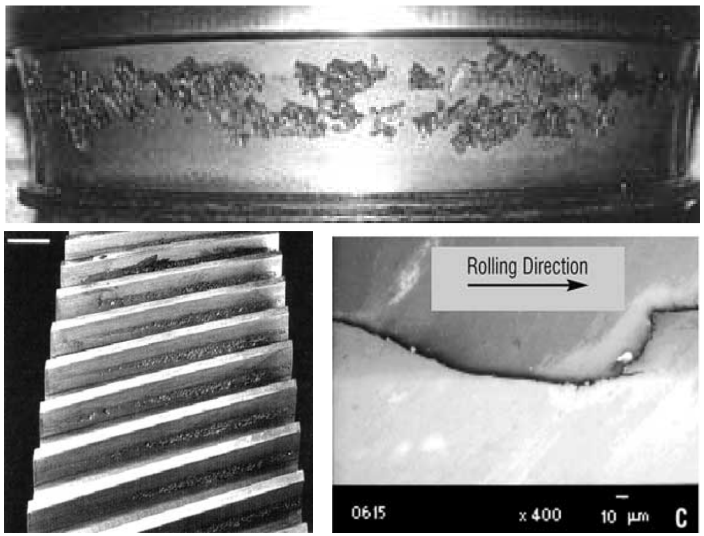
1. In-situ observation is a powerful technique to follow the evolution of void formation in dual phase steels.

2. In dual phase steels, voids in martensite dominate the behaviour because of the large number density and area fraction of voids and their early formation.

3. Control of microstructural parameters, such as hardness and volume fraction of martensite, can be used to control the void formation.

REWIND project

Knowledge-based engineering for improved reliability of critical wind turbine components



Coordinator: Jesper Hattel

Funding: Det Strategiske Forskningsråd (DSF)
Total : 30.1 mio. DKK + medfinan. 15,5 mio. DKK
MAC Wind: 3.7 mio. DKK

Partners: DTU Mekanik, Risø DTU, AAU-BYG,
HelmHoltz-Zentrum für Materialen,
Indian Institute of Technology,
DONG Energy A/S, Vattenfall A/S R&D,
Vestas A/S og MAGMA GmbH

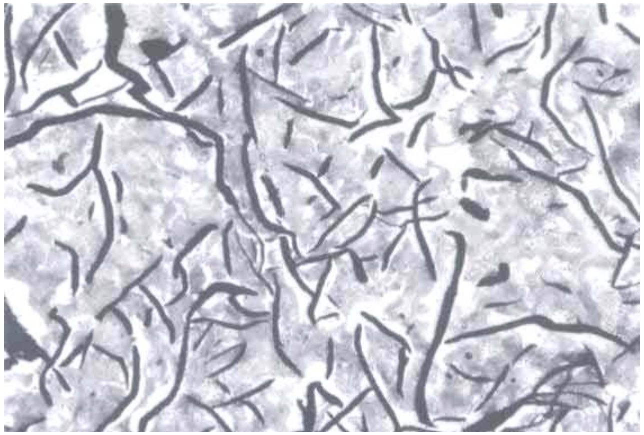
Material: Steel in drivetrain

Periode: 1 January 2011 – 31 December 2016
(MAC: 2011-14)

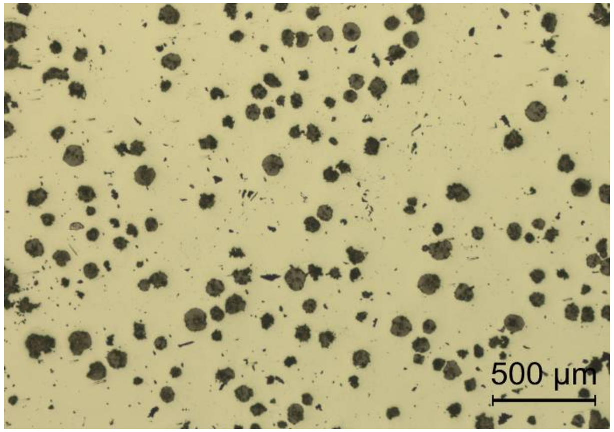
Failure analysis, defect classification, modelling of defects and material properties

Cheaper and more reliable wind turbines

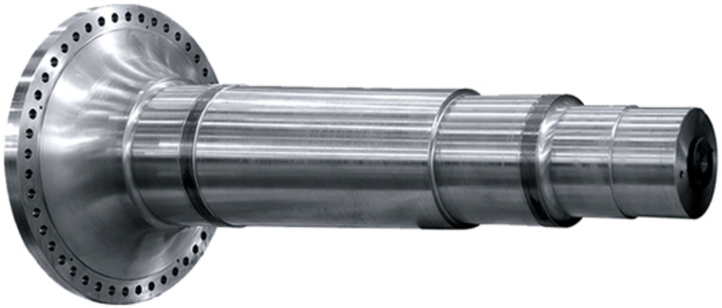
The structure of cast iron



Grey cast iron (graphite flakes)



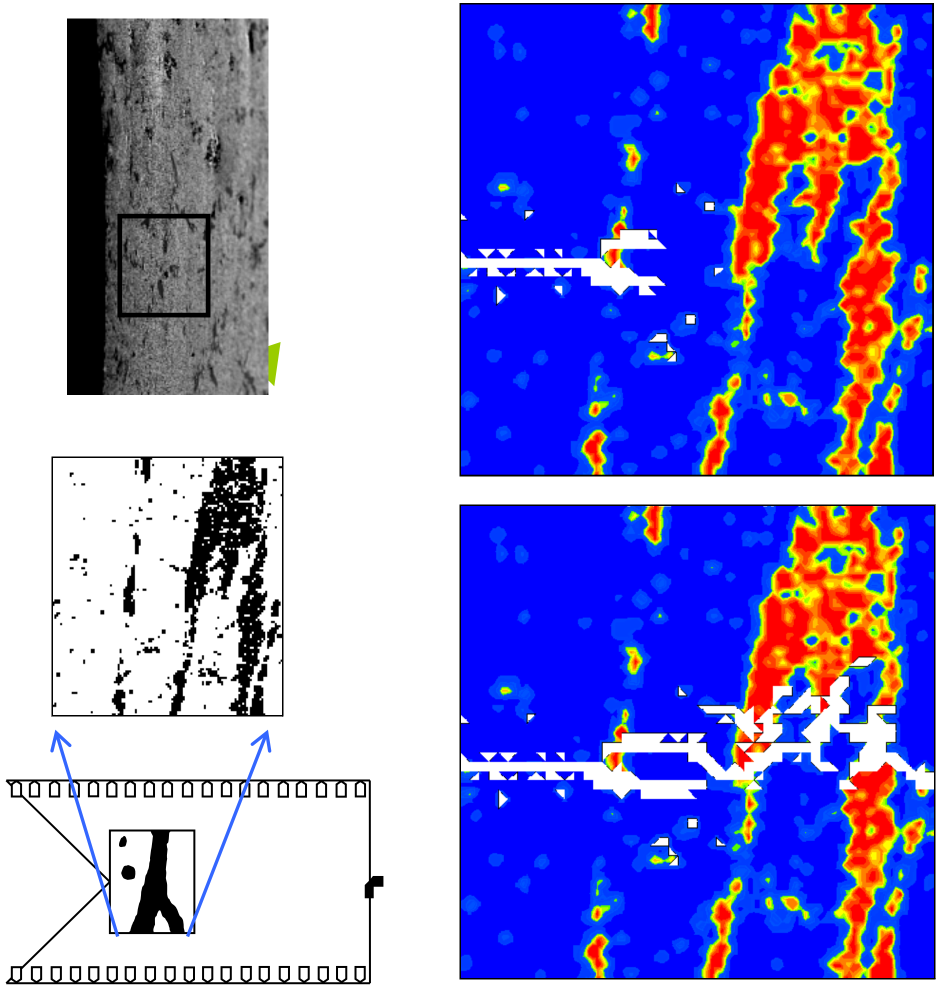
SG iron



Main shaft

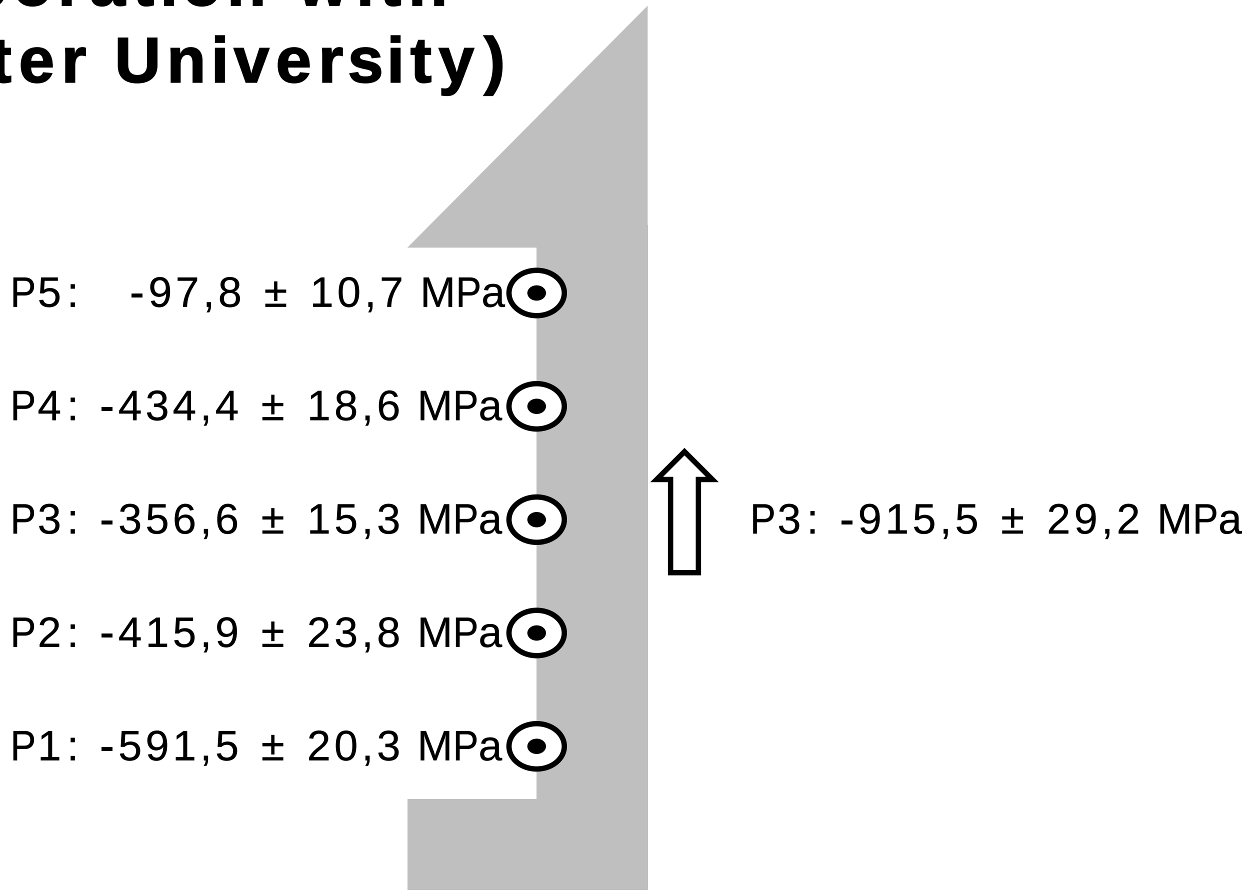
Size, shape and distribution of graphite nodules as input for FE models.

FE simulation of crack growth in the real microstructures



Distribution of inclusions and carbids

Laboratory X-rays Residual stresses (in collaboration with Manchester University)



Future research directions
Characterization, modeling and optimization

- Hard, wear and friction resistant materials including processing of surfaces
- Fatigue properties and fatigue resistant materials including processing of surfaces
- Failure and damage: Non-destructive 3D characterization using x-ray tomography of structures and structural defects (components - micrometer – nanometer scales)
- Residual stresses – in combination with microstructural investigations to underpin analysis of failure mechanisms

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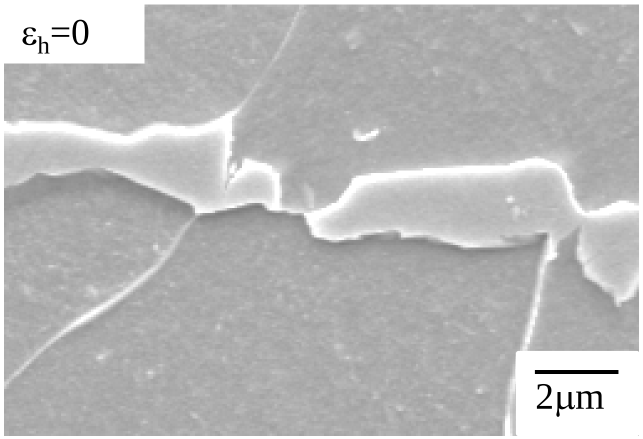
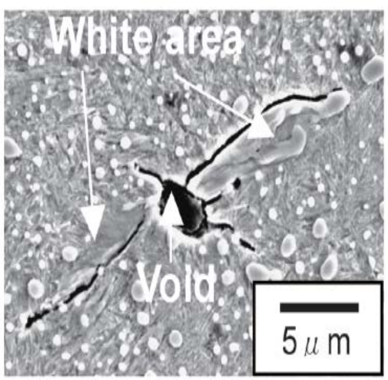
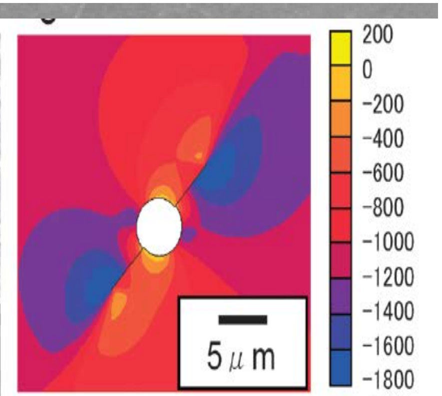
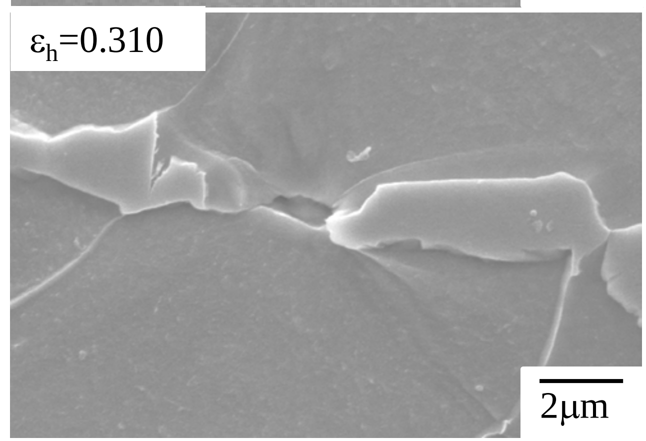
- Welding : welding processes, effects of welding on microstructure, voids/cracks
- Corrosion: in-situ observations, effects of microstructure on corrosion

Concluding Remarks



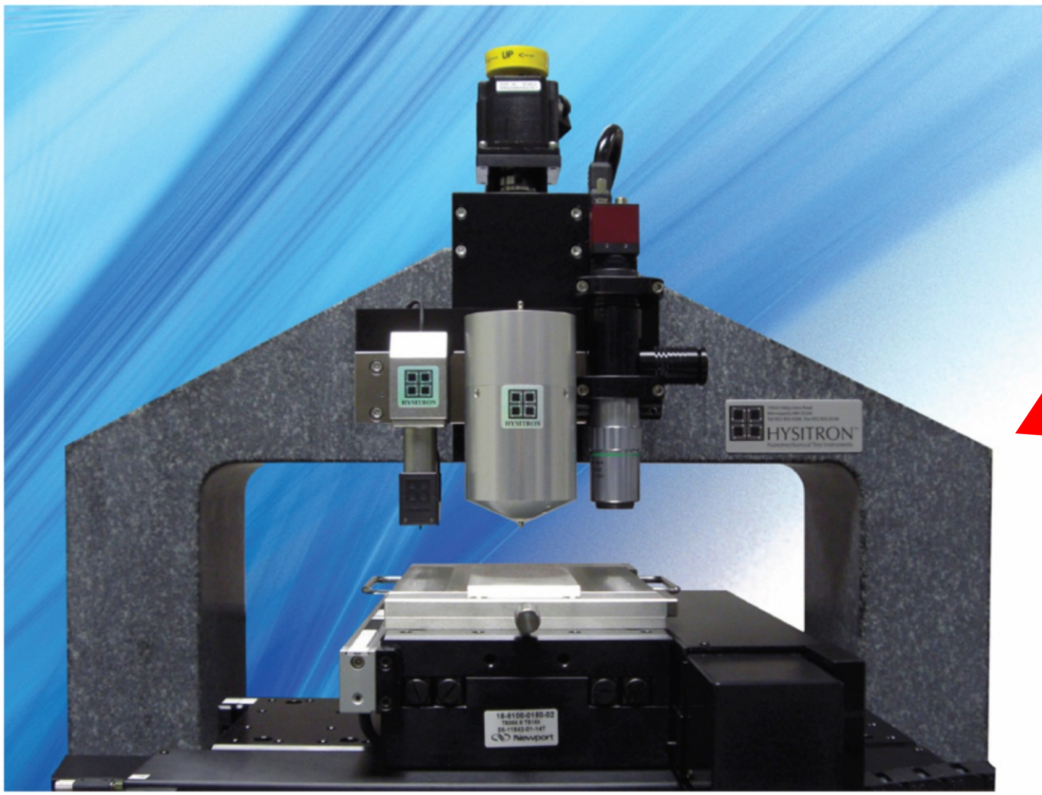




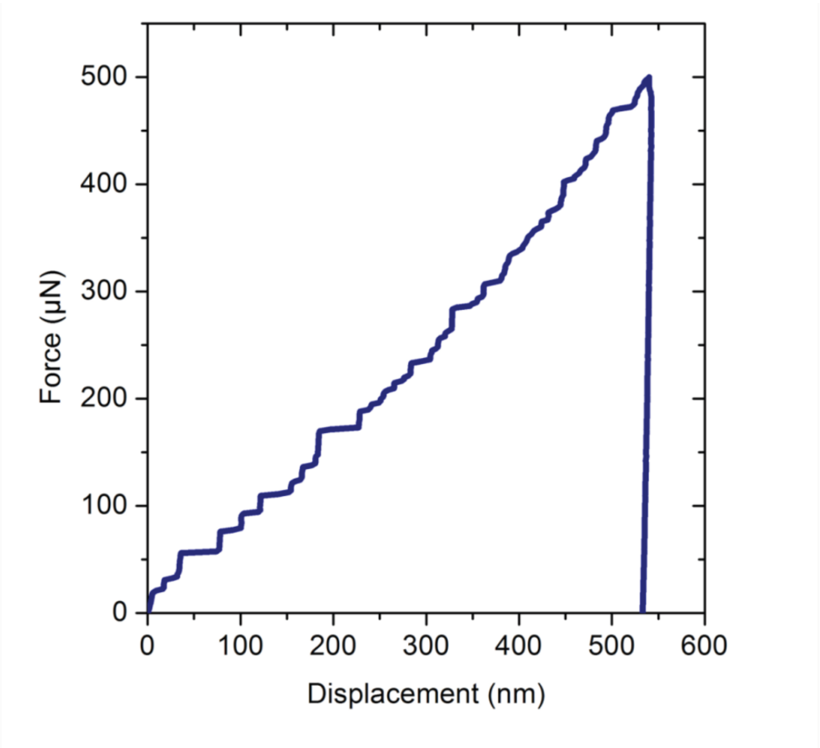


Nanoindentation

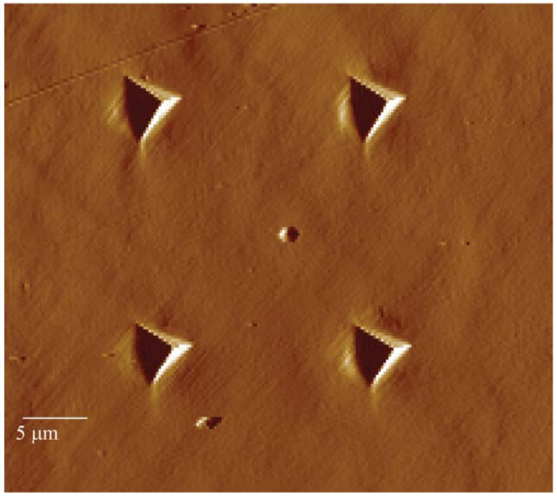
In-situ imaging with nanoindentation
and
nanomechanical property measurement capabilities



Load-displacement curve and SPM



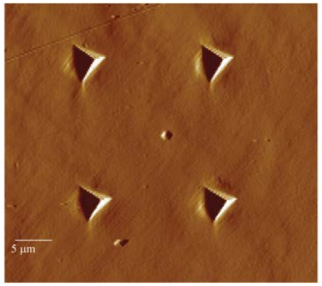
Load-controlled nanoindentation test on single crystal (100) Al showing dislocation activity throughout the testing cycle



Four indents in copper as imaged by *in situ* SPM imaging mode

Scanning probe microscopy (***SPM***)

Hardness Mapping



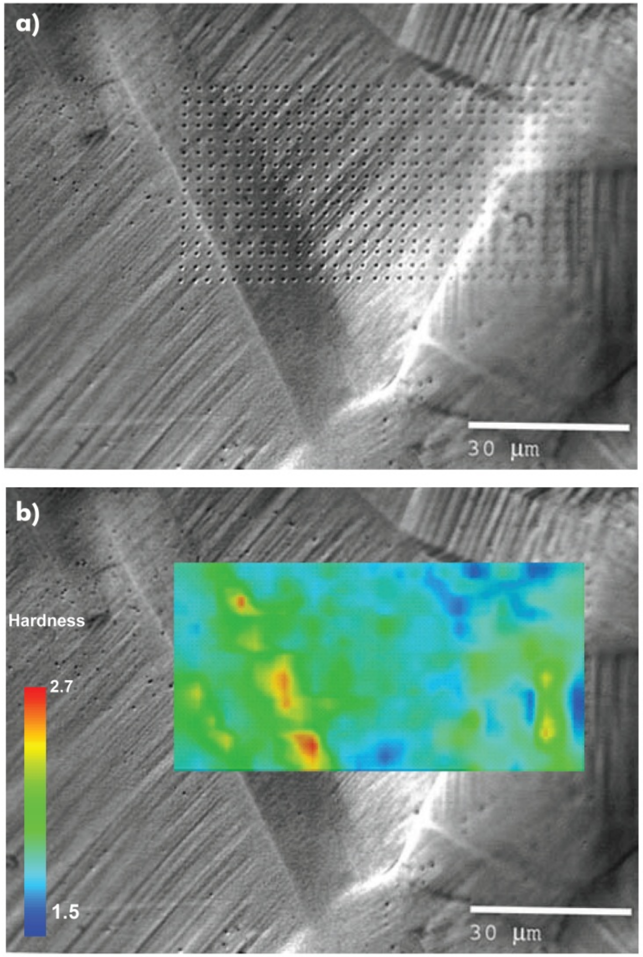
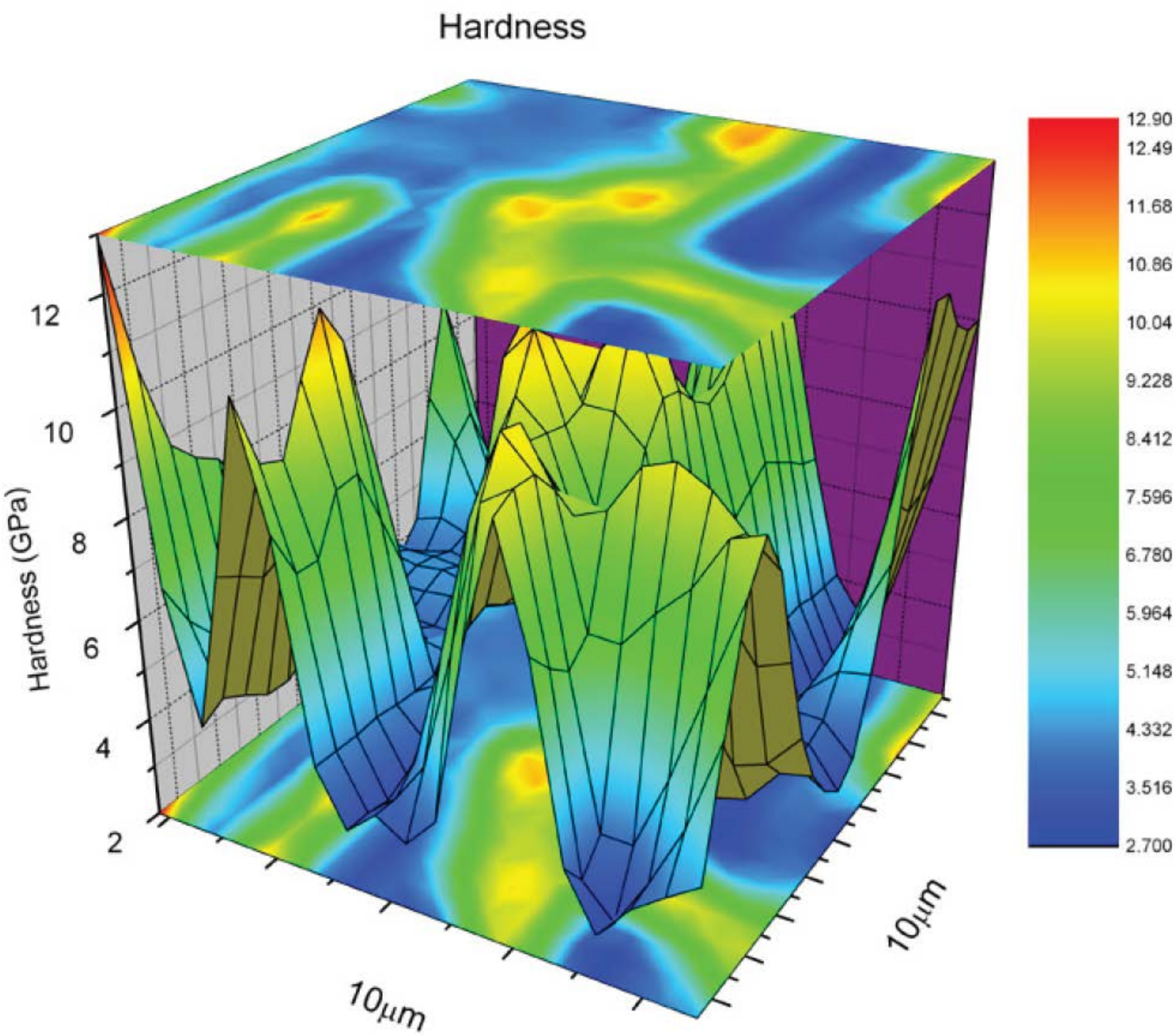


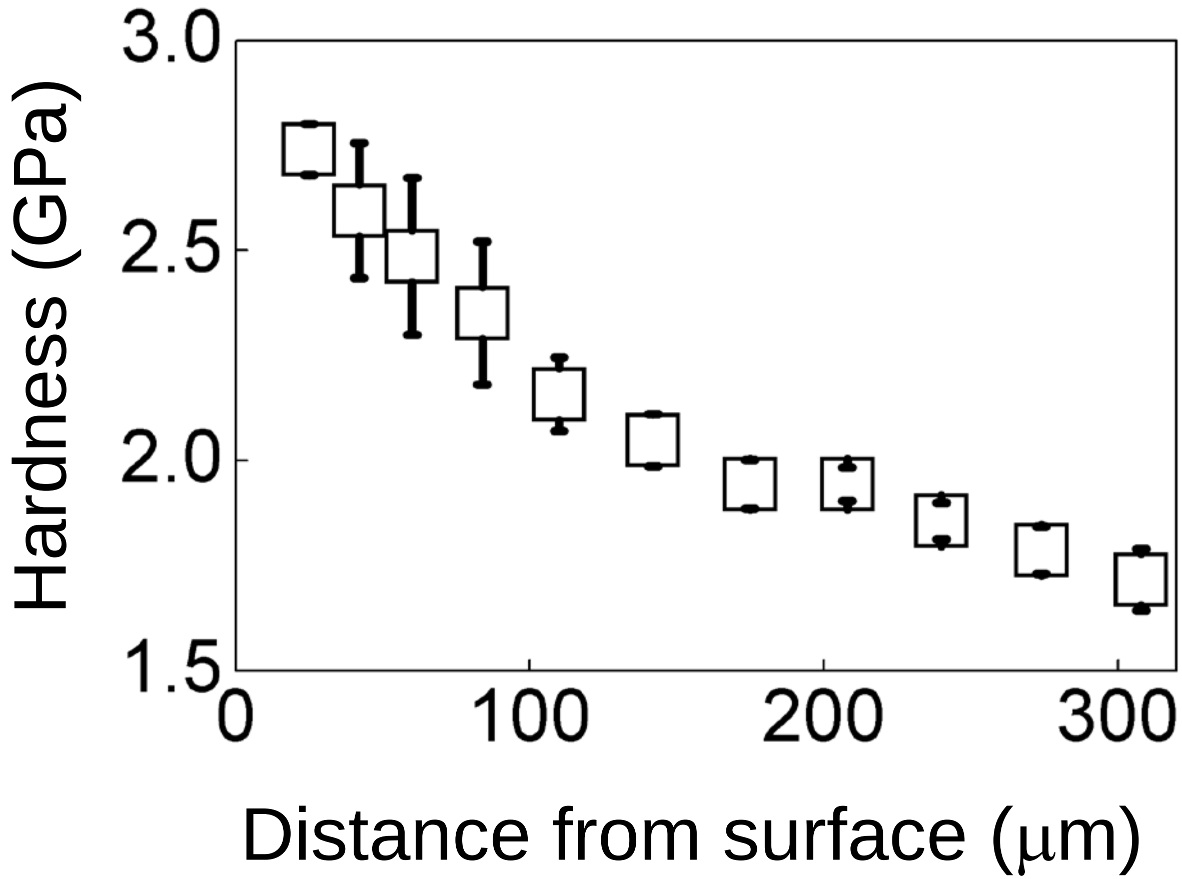
Figure 2: a) Optical micrograph of a grid of **TriboIndenter** indents superimposed on a twin and grain boundaries in Copper strained to 5%. (b) Corresponding hardness map indicates that the twin boundary is locally harder than the matrix material.



: Hardness map showing distinct ferrite and martensite phases.

Microhardness

The application of microhardness testing with fairly large indents did only measure the hardness up to about 25 μm from the surface, which with reference to the figure exclude the hardest part of the structure.



Scanning wear

Measuring wear resistance at the nanoscale

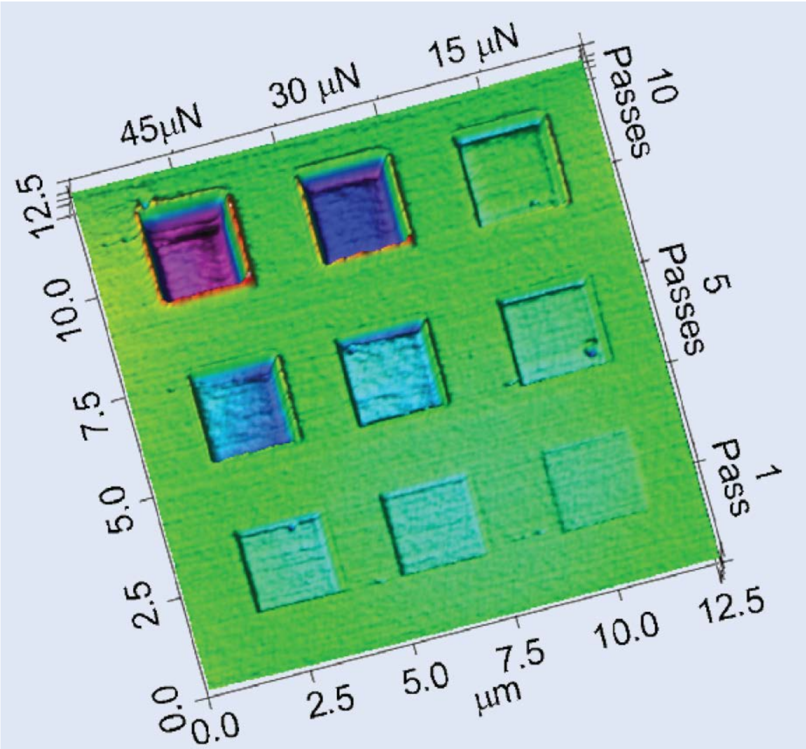
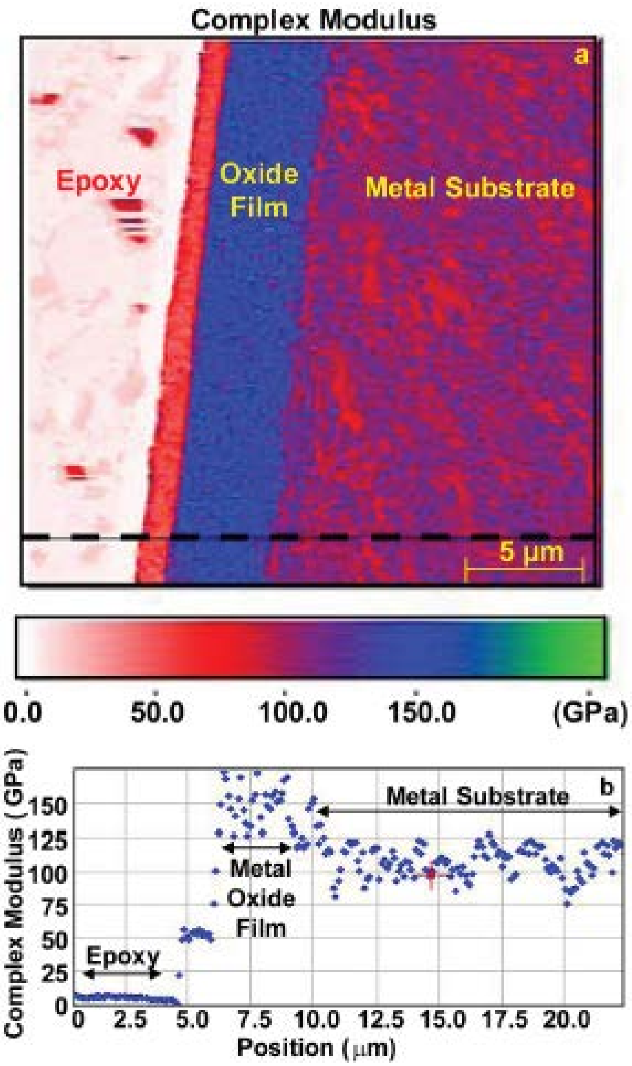
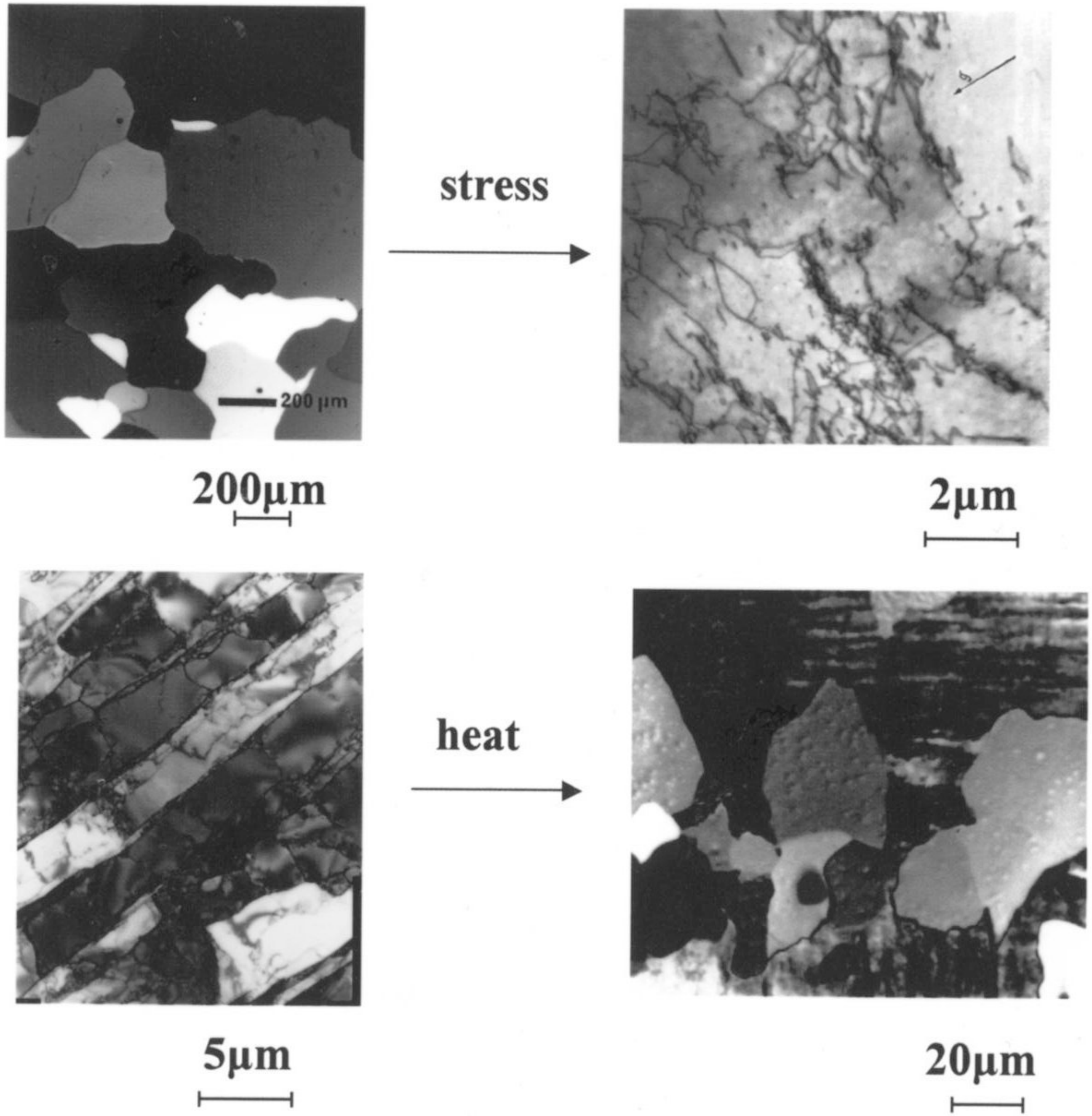


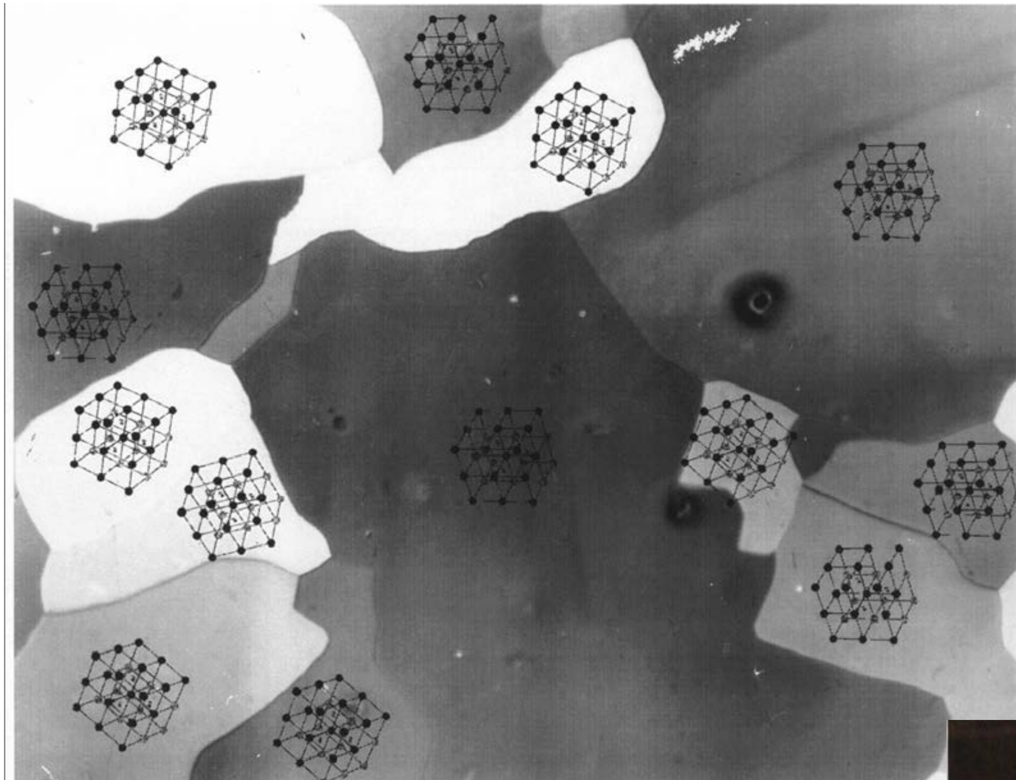
Figure 1. **ScanningWear** test on DLC (Diamond-Like Carbon) coating on a computer hard disk drive with 15 μN, 30 μN and 45 μN loads and 1, 5 and 10 passes respectively.

Modulus Mapping

of a Metal Oxide Film

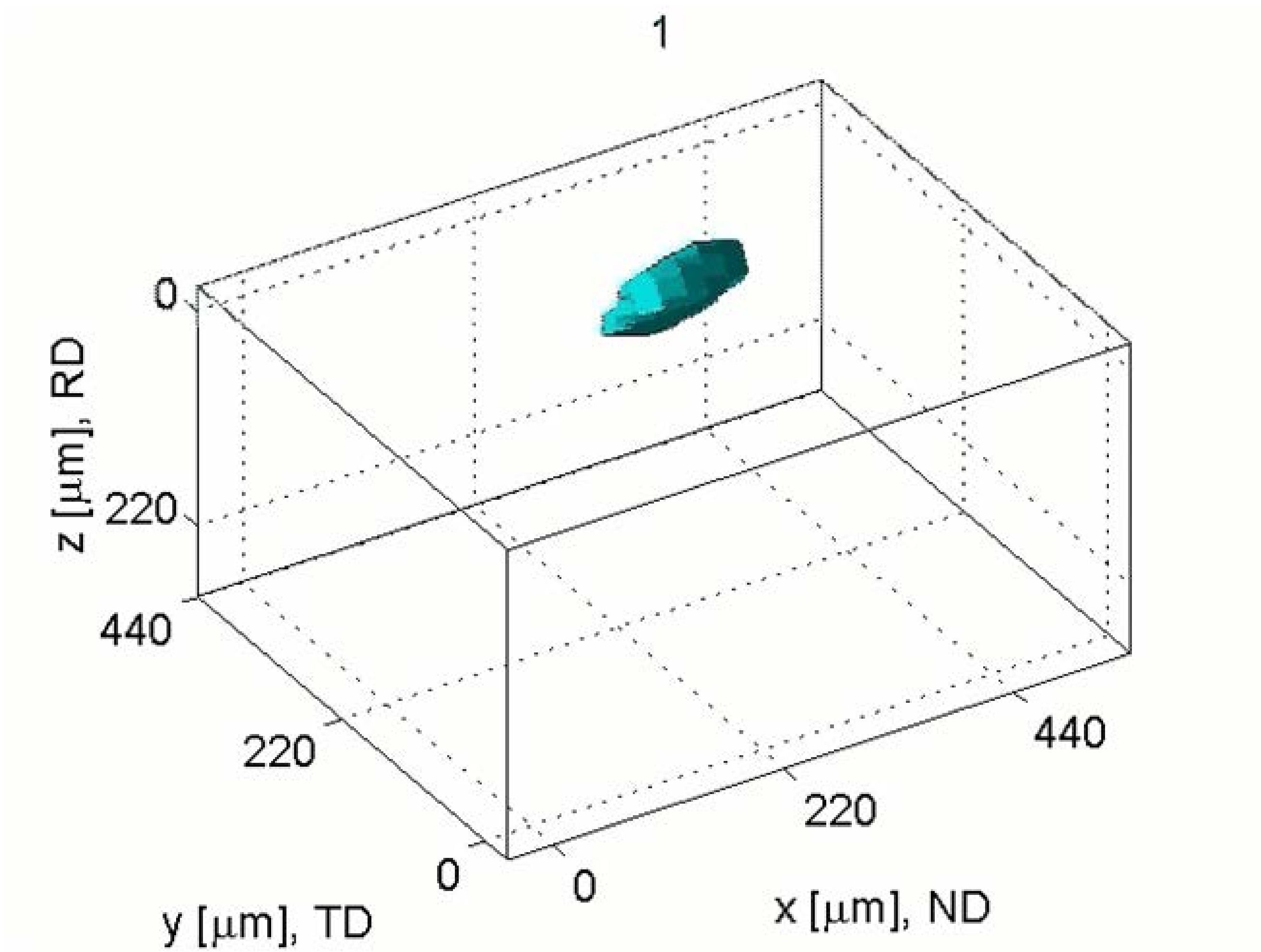



200 µm
stress
200µm
2µm
heat
5µm
20µm



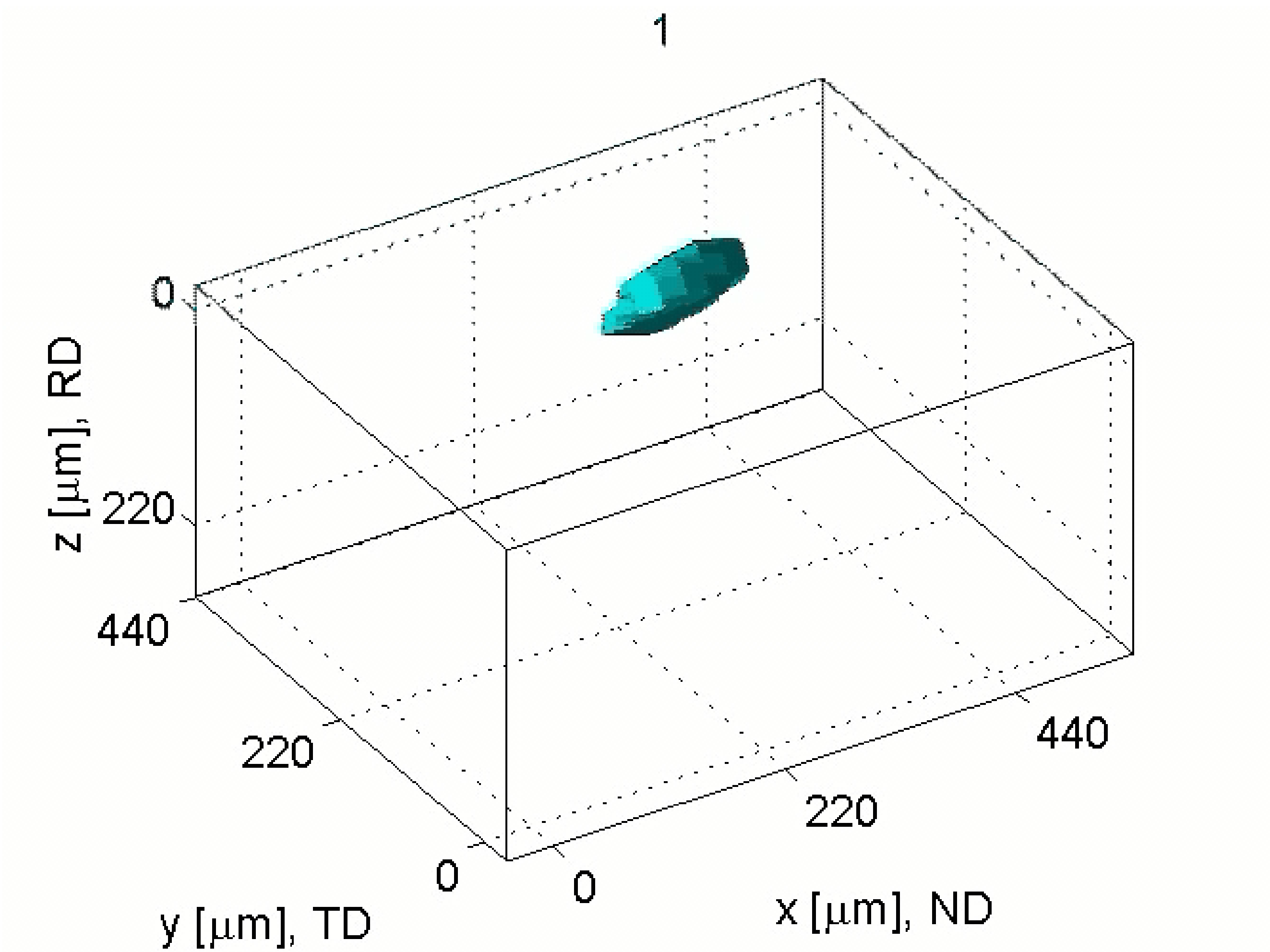

Coca-Cola
Export
FINSPONG
ALUMINIUM

Grain growth during recrystallization in weakly rolled aluminum single crystal



Schmidt, S., Nielsen, S.F., Gundlach, G., Margulies, L., Huang, X., Juul Jensen, D., Science, 2004, 229-232.

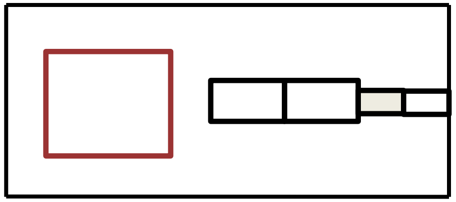
Grain growth during recrystallization in weakly rolled aluminum single crystal

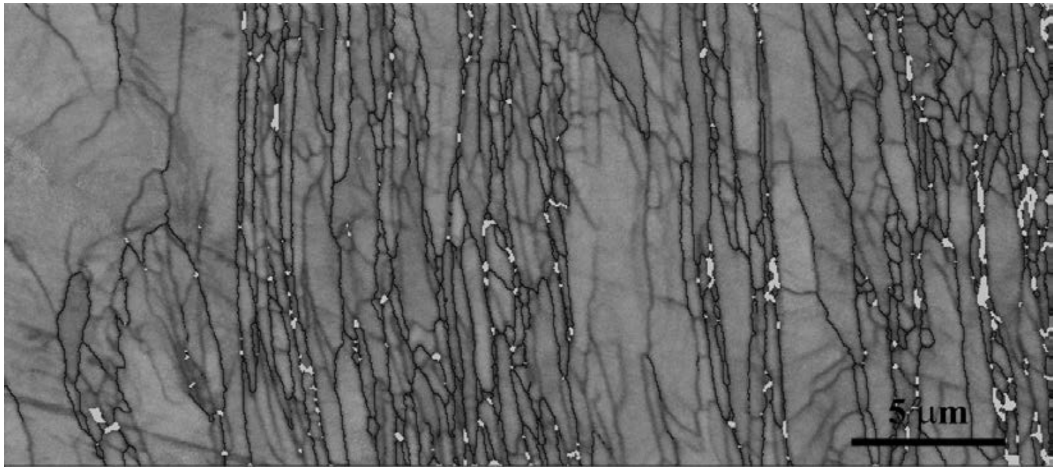


Schmidt, S., Nielsen, S.F., Gundlach, G., Margulies, L., Huang, X., Juul Jensen, D., Science, 2004, 229-232.

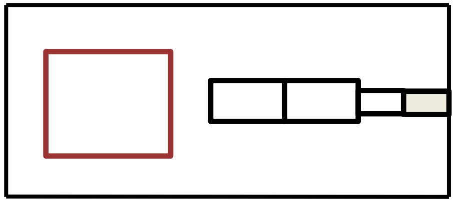
Shot-peening

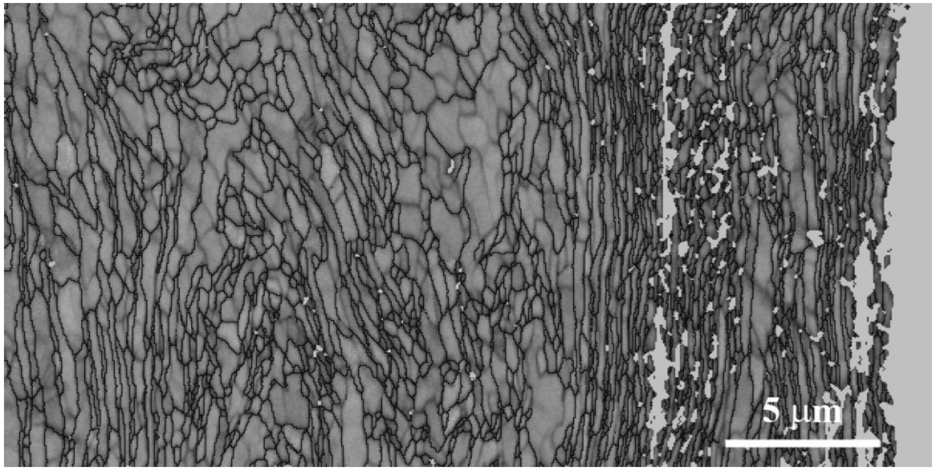
e: 60 - 30 μm from surface





f: 30 - 0 μm from surface





Inner surface

Black line: high angle boundary (Misorientation angle > 15°)

Hard and wear resistant steel components

- Characterize structure to determine stress and strain gradients as input for numerical modelling of e.g. friction and wear
- Develop reliable testing techniques (e.g. microsamples) to analyse structure and properties of components damaged by impact, wear or fatigue

Light and strong metals and alloys

- Optimize strength and formability by thermomechanical processing – bulk samples and multilayers
- Advance analytical and numerical modelling of receovery and recrystallization through 2D and 3D characterization

Technique development

- Implement and develop techniques for characterization of damaged samples
- Develop techniques for optimizing metals including surface hardening

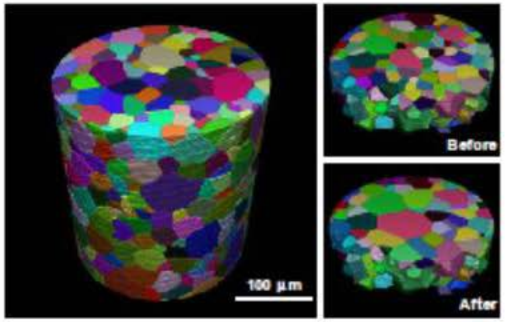
Reliability

- Materials parameters into the modeling of drive train components
- Effects of local inhomogeneties

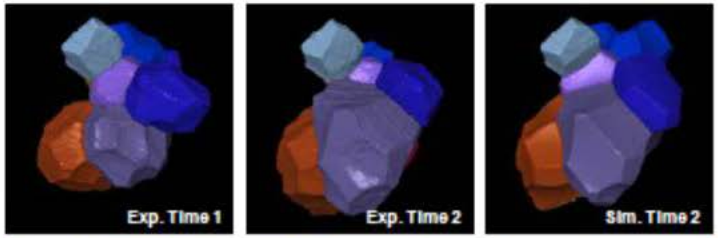
Grain Growth Kinetics in Titanium

High fidelity 3-D synchrotron imaging techniques and robust phase field modeling allow capture of anisotropy effects

- Development of advanced non-destructive 3-D imaging tools based on phase-contrast X-ray tomography and diffraction contrast X-ray tomography.
- Acquisition of large (1000+ grains) data sets providing direct insight to the grain evolution in Ti-β-21S during grain growth.
- Developed a 3-D anisotropic phase field model for cubic materials.
- Simulated grain evolution in a Ti-β-21S sample with over 1200 grains.
- By comparing the morphologies of individual grains predicted by simulation with those measured experimentally we find that:
  a) The morphological evolution of a grain depends only on its local ensemble of grains.
  b) The mobilities of a grain's boundaries can vary by orders of magnitude and depend strongly on the grain boundary normal.
  c) Even without accounting for anisotropy in the grain boundary energy the model is capable of predicting surprisingly accurate morphologies and topologies in the isotropic regions of the experimental dataset.



3-D experimental data of Ti-β-21S.
Left: Initial structure used in the phase field simulation comprising more than 1200 grains.
Right: Subset showing the grain structure before and after grain growth, respectively.



Comparison of an ensemble of grains in simulation with experiment

MAC - Materials Science and Advanced Characterization section in DTU WIND ENERGY

Senior researchers	4
Researchers	3
Engineers	0
Postdocs	2
PhD students	4
Technicians	3
Secretary	1
Guest scientists	3

Close collaboration with KOM

2012	
Journal papers	16
Conference papers	30
Books	1

Externally funded projects (MAC)

- Danish-Chinese Center for Nanometals
- Nippon Steel collaboration contract
- BladeKing (KOM)
- Rewind (MEK)
- Armour Altia (KOM)
- Wear in Rails
- Materials for fusion (MEK)
- ViNaT (KOM)
- New Electron microscope

Danish-Chinese Center for Nanometals 2009-2015 25mil DKK (DTU)

MAC

Shenyang National Laboratory (CAS)

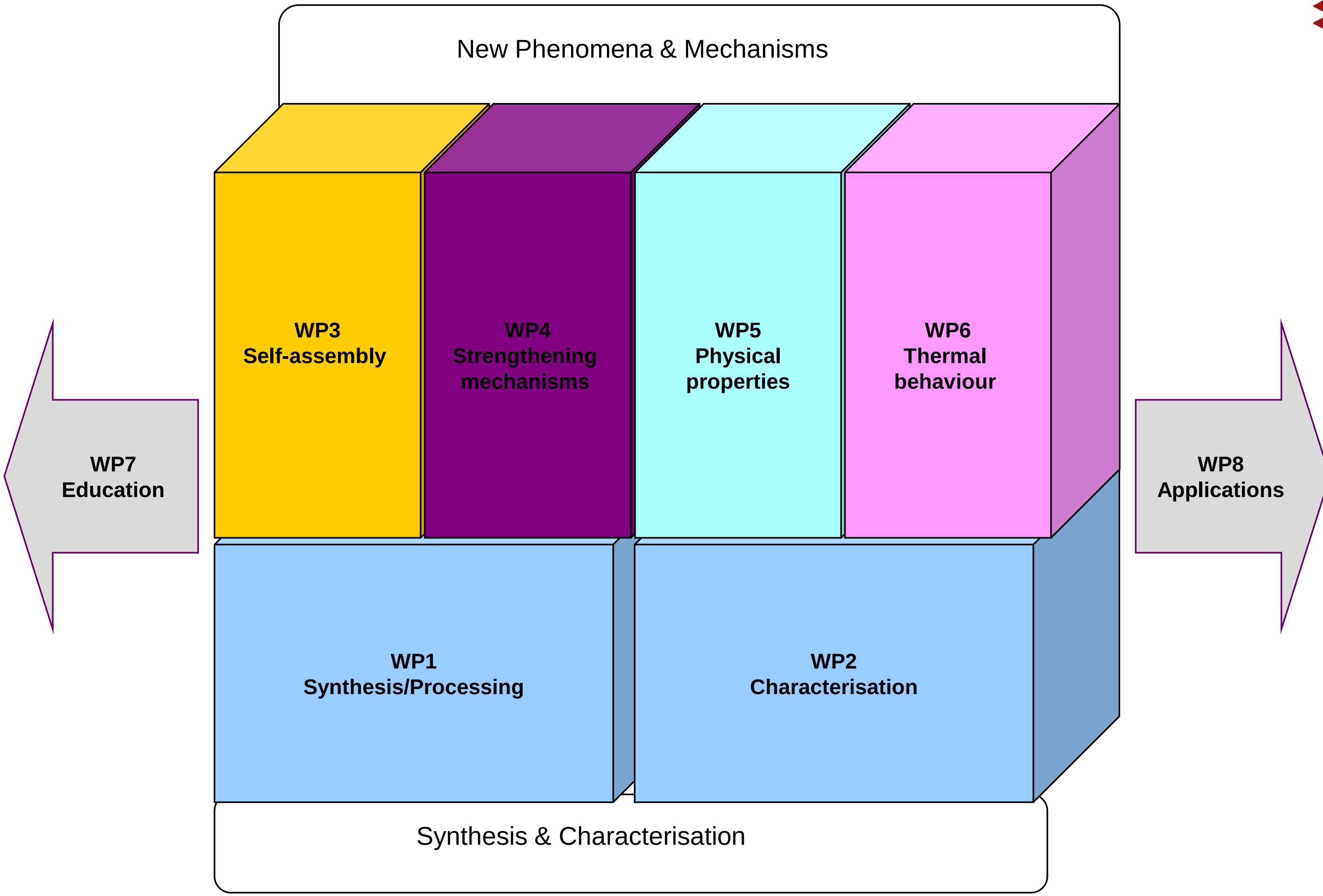
FUNDED BY

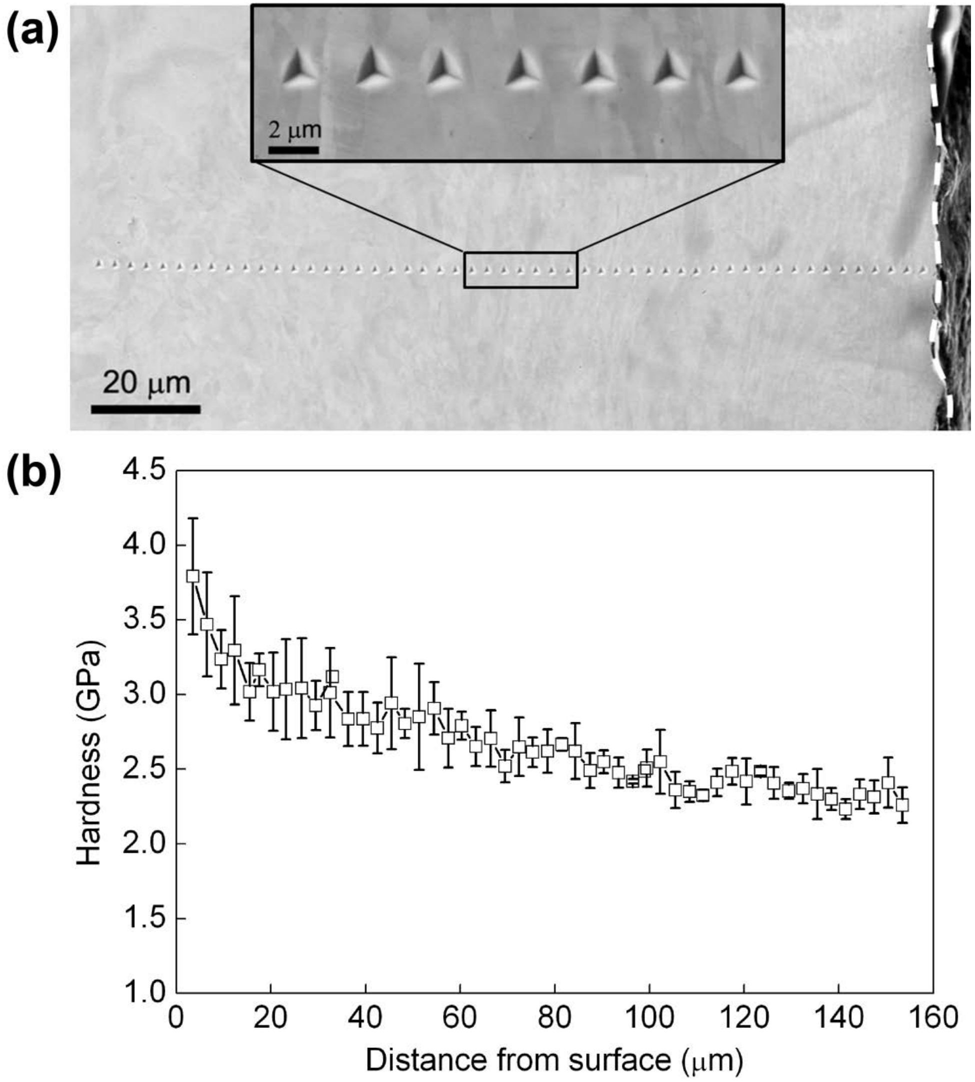
Danmarks Grundforskningsfond
Danish National Research Foundation

NSFC

Tsinghua University

Chongqing University


New Phenomena & Mechanisms
WP3
Self-assembly
WP4
Strengthening mechanisms
WP5
Physical properties
WP6
Thermal behaviour
WP7
Education
WP8
Applications
WP1
Synthesis/Processing
WP2
Characterisation
Synthesis & Characterisation



Nanohardness approaching the surface of peened specimen: (a) SEM micrograph showing one line of the nanoindents and (b) nanohardness versus distance to surface. The white dashed line in (a) shows the position of the surface.

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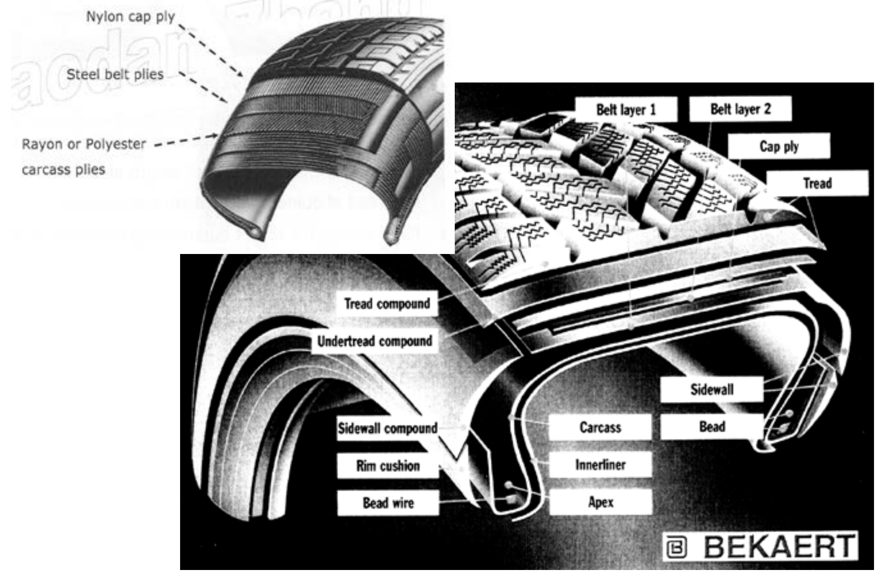
Cold drawn high-carbon steel wires

---- highest strength of all mass-produced steel products, > 5GPa

Cables for suspension bridges

Springs

Steel cords for automobile tires



Potential use in wind mills

BladeKing

REWIND

Knowledge based engineering for improved reliability of critical wind turbine components

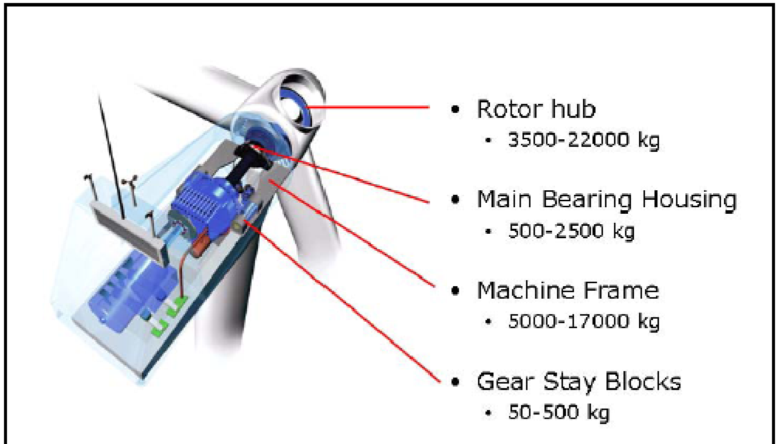
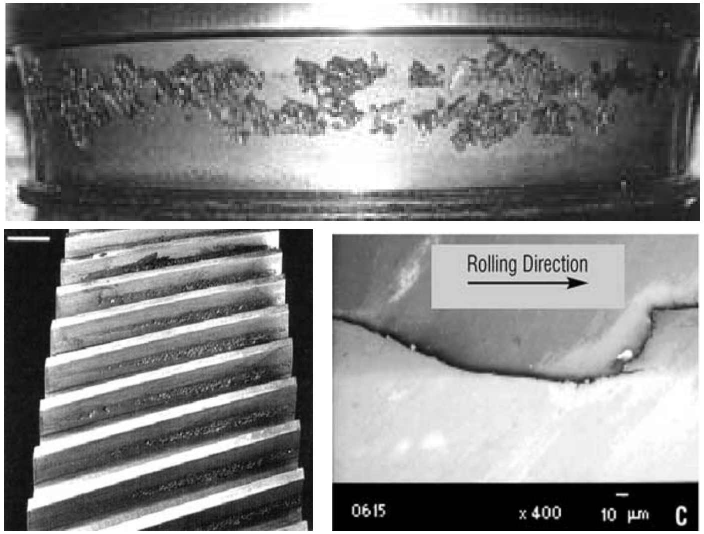


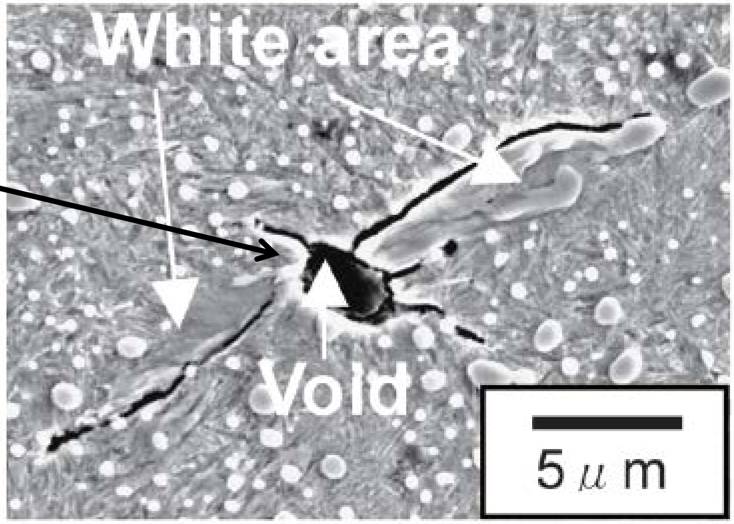
***Figure 2**: The main cast parts inside an NM44 nacelle*

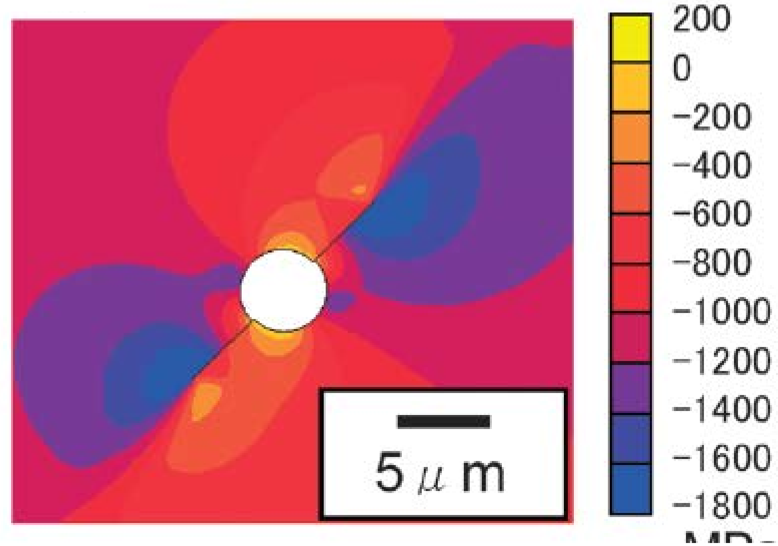


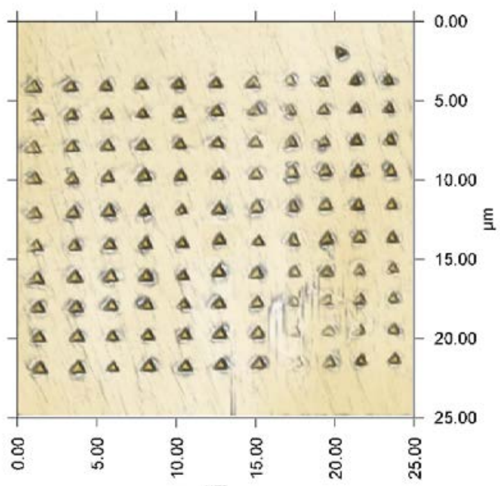
Pitting, flaking, spalling and cracking

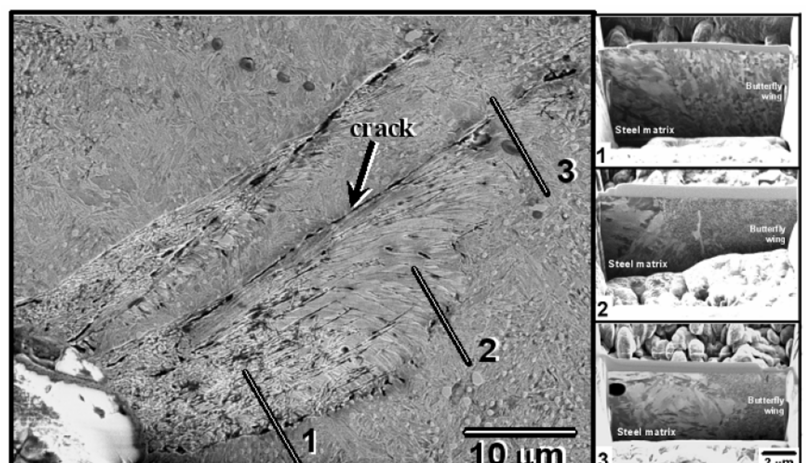
Butterflies and WEA

EDS
EELS









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- Materials for fusion (MEK)
- ViNaT (KOM)
- New Electron microscope

On-going major collaborations

Universities

- Leuven, Belgium
- Ecoles de Mines, France
- Manchester, UK
- Cambridge, UK
- Leoben, Austria
- Ghent, Belgium
- NTNU, Norway

- Oak Ridge, USA
- Sandia National Laboratory, USA
- Argonne National Laboratory, USA
- Berkley, USA

- Tsinghua, China
- IMR Shenyang, China
- Chongqing, China
- Kyoto, Japan

Industry

- Bekaert, Belgium

- Tata Steel, India, The Netherlands, UK

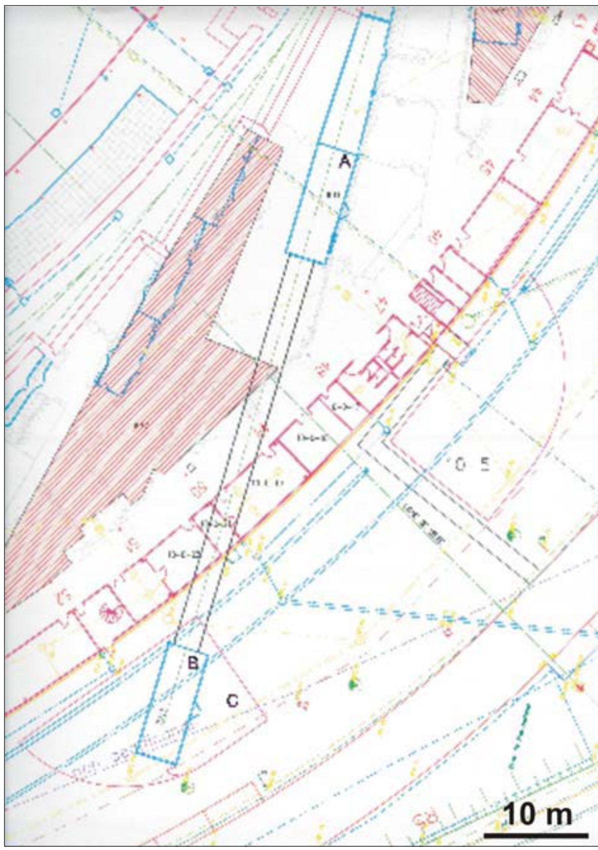
- Nippon Steel and Sumitomi Group, Japan
- Baosteel, China
- Fasten Group, China

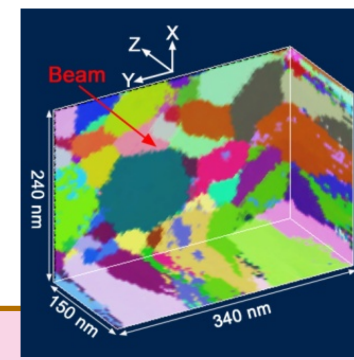
DTU

- MEK
- FYS
- CEN
- IMM
- Nanotek

3D X-ray microscopes now also at APS in USA, SPring 8 in Japan and Hasylab in Germany

ESRF in France: 3D X-ray nanoscope





3D X-ray　　3DTEM

cm　　100nm　　<10nm

Hard and wear resistant steel components

- Characterize structure to determine stress and strain gradients as input for numerical modelling of e.g. friction and wear
- Develop reliable testing techniques (e.g. microsamples) to analyse structure and properties of components damaged by impact, wear or fatigue

Light and strong metals and alloys

- Optimize strength and formability by thermomechanical processing – bulk samples and multilayers
- Advance analytical and numerical modelling of receovery and recrystallization through 2D and 3D characterization

Technique development

- Implement and develop techniques for characterization of damaged samples (incl lab residual stress measurements)
- Develop techniques for optimizing metals including surface hardening
- Superusers of all relevant 3D/4D techniques with focus on research results

Initiated new collaborations

- Delft University
- Inst. für Eissenf. Düsseldorf
- Aachen University

- LORC
- Force
- Siemens Wind Power

New

Equipment

- Lab x-ray tomography
- Lab x-ray residual stress
- Nanometal processing and testing
- (Atom probe microscope)

Staff increase

1-2 senior researcher

1-2 researchers

1 development engineer

1-2 technician

4-5 PhDs and PDs

My own research areas

- Spatial and temporal variability and effects hereof on performances of materials
- Crack initiation and growth – relations to microstructures (use 4D methods)
- Research output from 3D and 4D methods

Future research directions
Characterization, modeling and optimization

- Hard, wear and friction resistant materials including processing of surfaces
- Fatigue properties and fatigue resistant materials including processing of surfaces
- Failure and damage: Non-destructive 3D characterization using x-ray tomography of structures and structural defects (components - micrometer – nanometer scales)
- Residual stresses – in combination with microstructural investigations to underpin analysis of failure mechanisms

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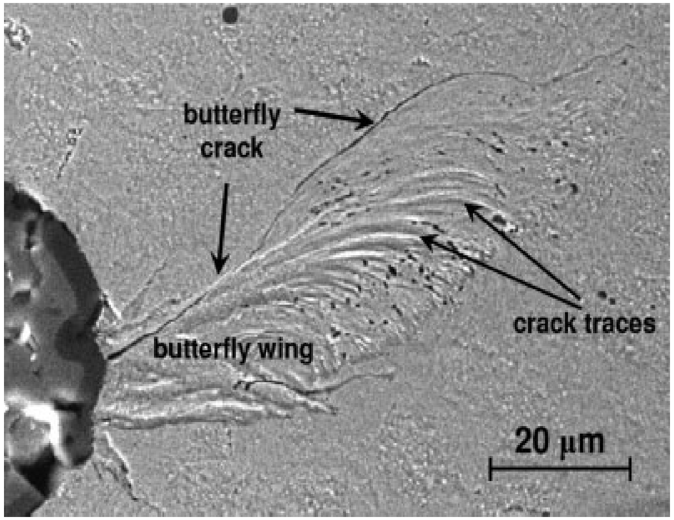
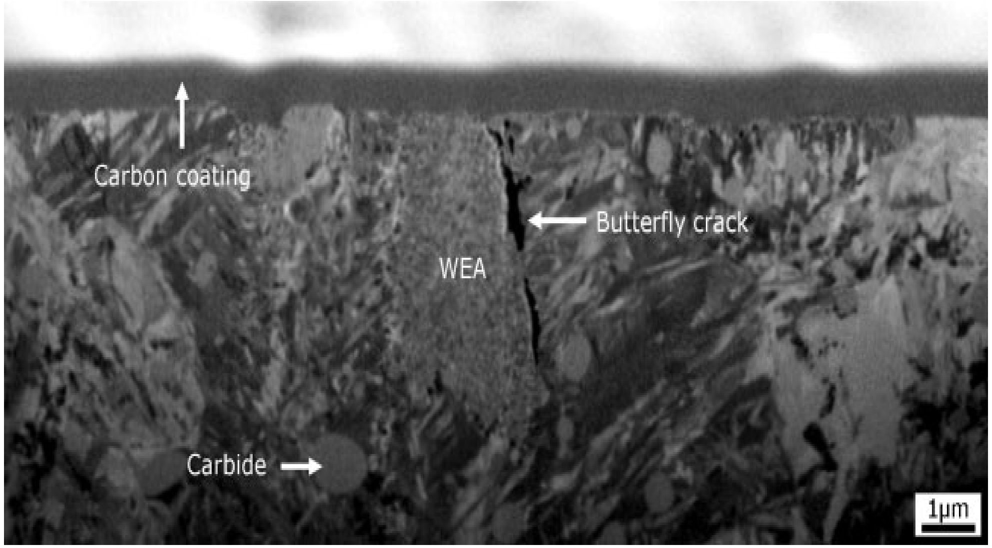
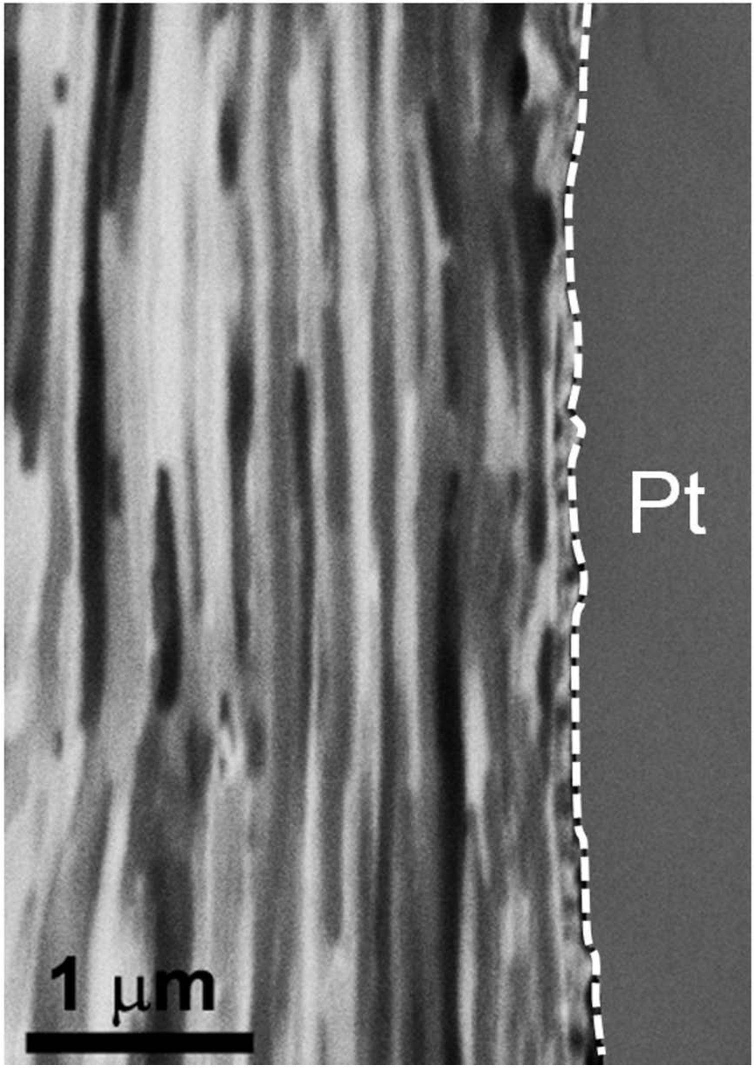
- Welding : welding processes, effects of welding on microstructure, voids/cracks
- Corrosion: in-situ observations, effects of microstructure on corrosion

Butterfly cracks in bearings

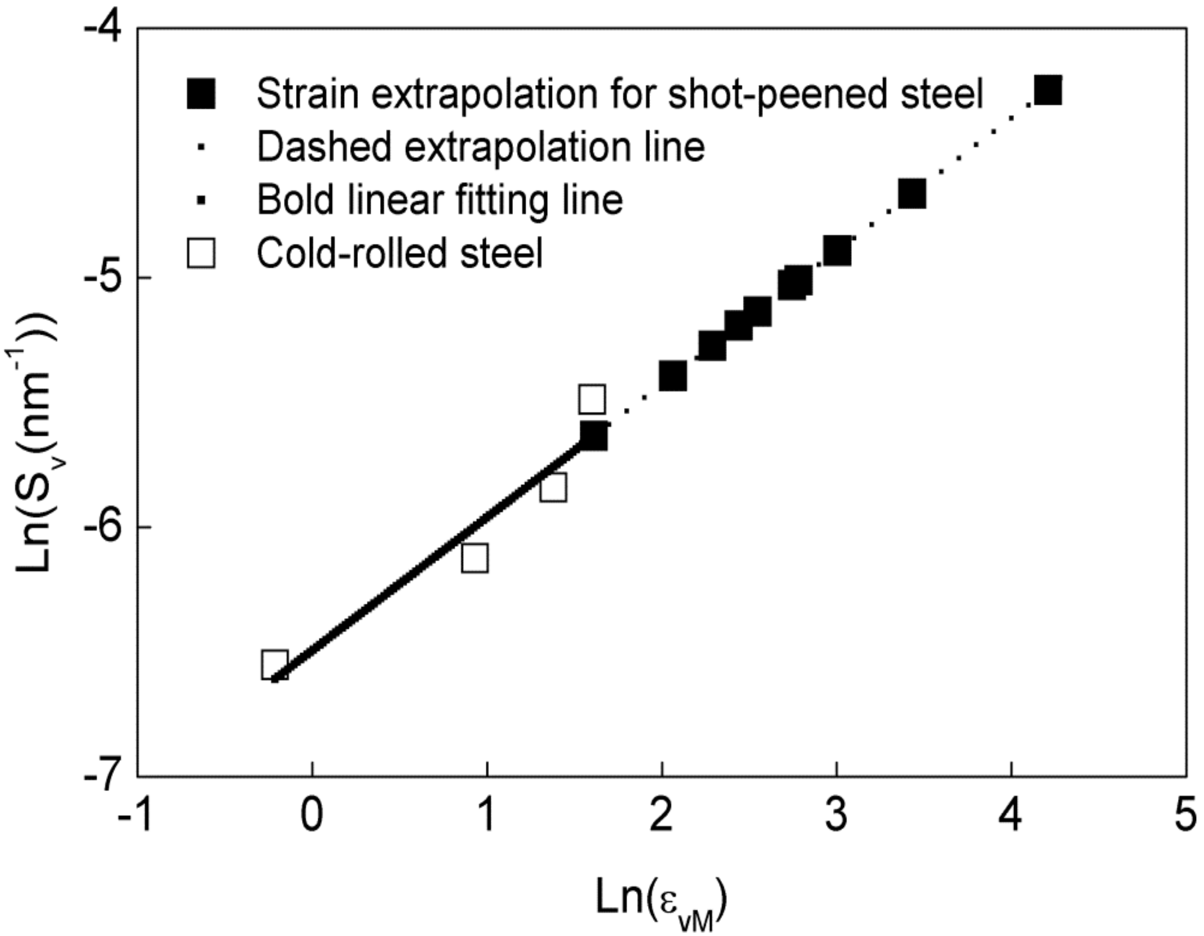




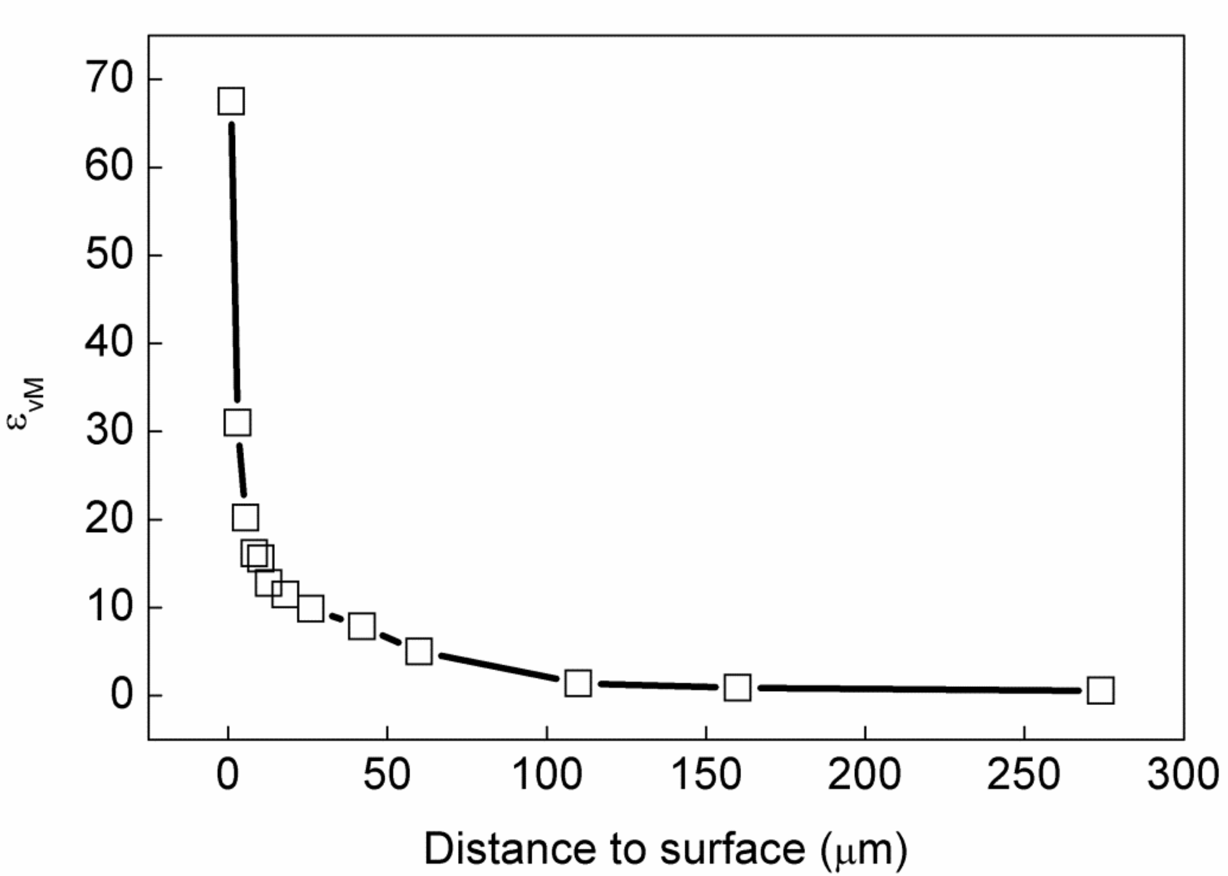
SEM image of butterfly crack starting from an inclusion and a cross section through a butterfly crack made by a FIB.



Microstructure of the surface layer in the shot-peened specimen cut by focused ion beam with the surface protected by deposited platinum. The white dashed line shows the position of the peened surface.

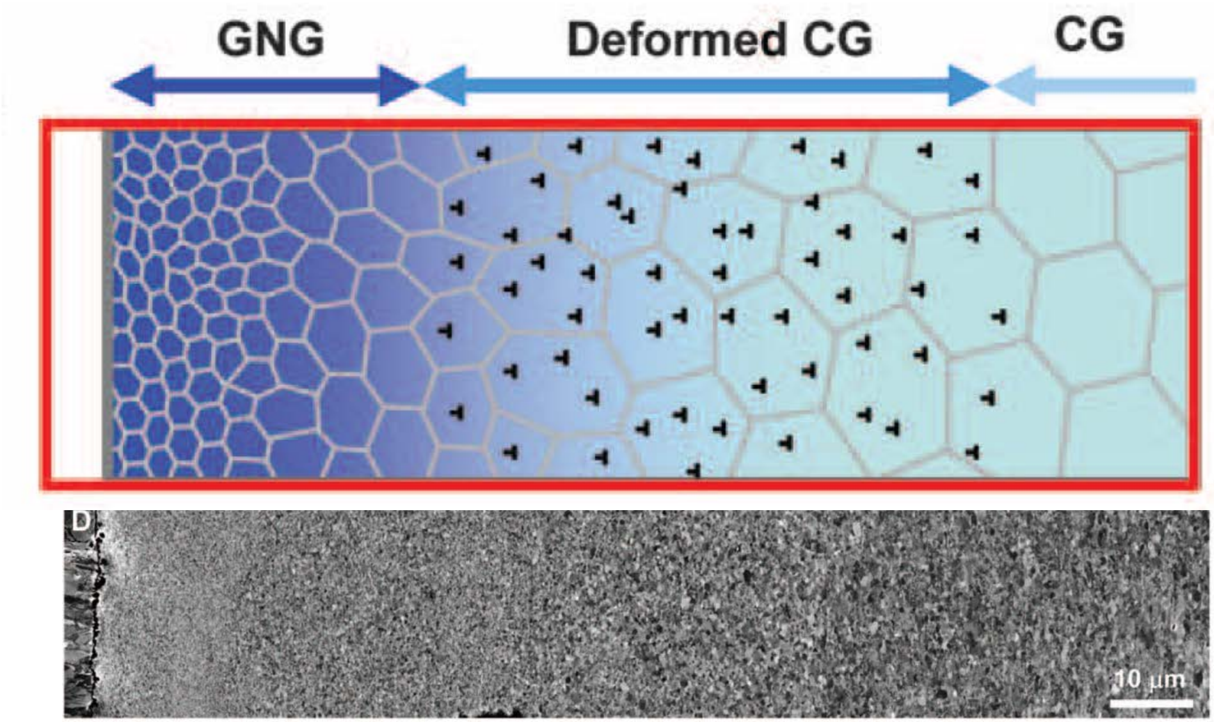


Power law relationship between boundary area per unit volume (S_v) and von Mises strain (e_{vM}) for the steel deformed by cold rolling and shot peening.

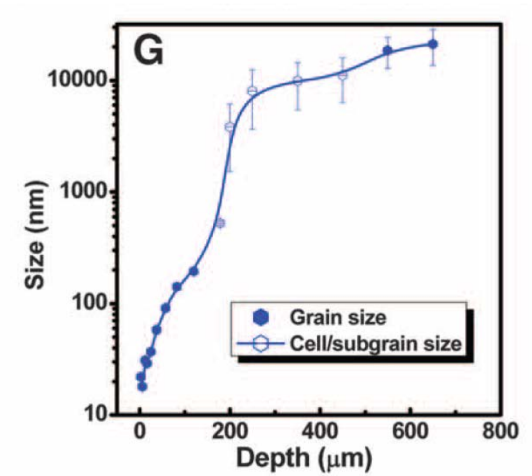


ε_{vM} as a function of distance from surface of peened steel

Processing of Surfaces
Graded structure in Cu
produced by surface plastic deformation



GNG: graded nanograins
CG: coarse grains



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Aim (MAC)

To perform materials science and development on a high international level with focus in particular on materials and components for wind energy applications

To advance existing techniques and to implement new characterization techniques and data analysis tools to match the needs of the scientific and engineering projects

Covering the whole range from basic science to applications

Work on length scales from nanometer to meter